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JULY 1950

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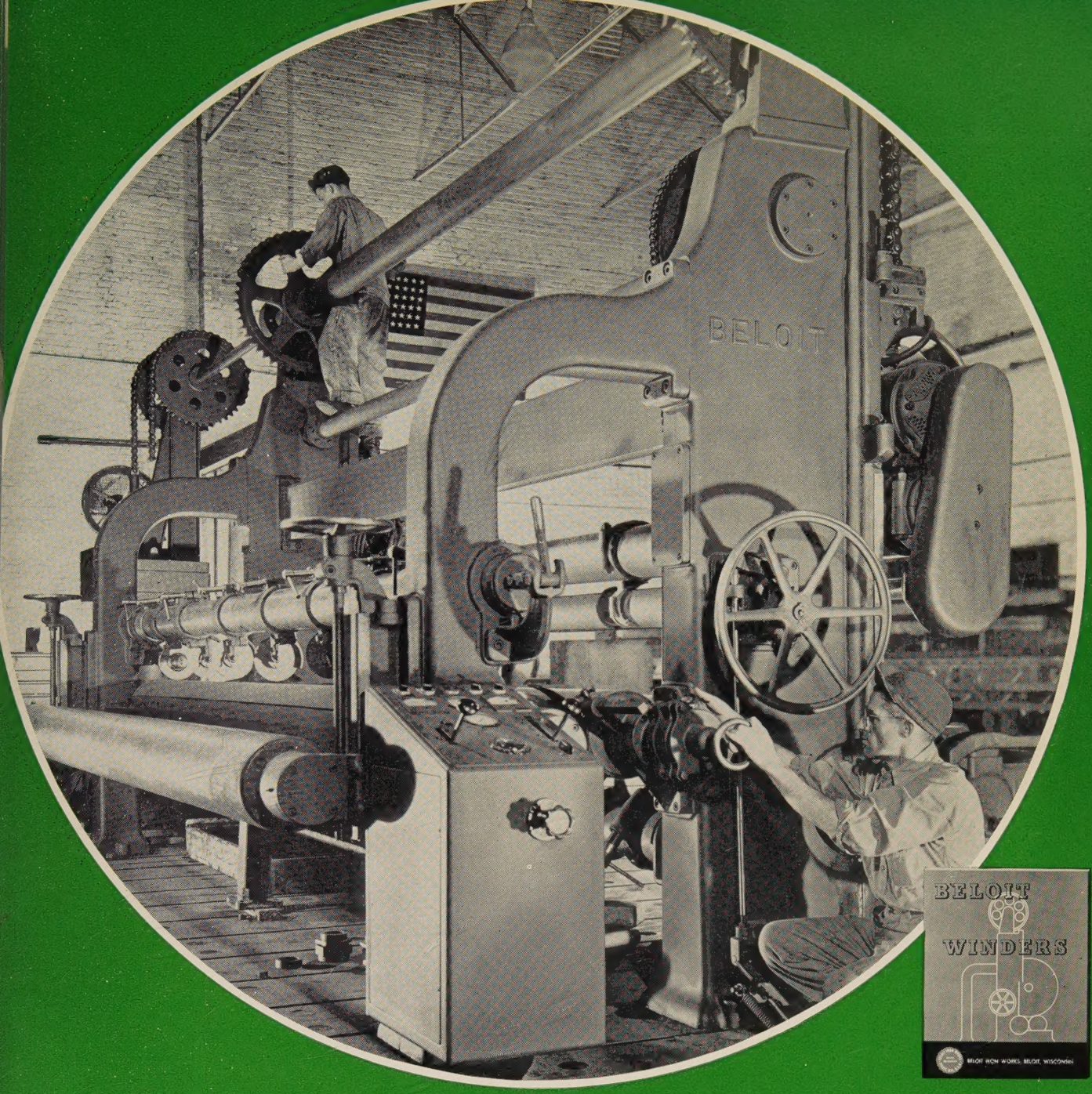

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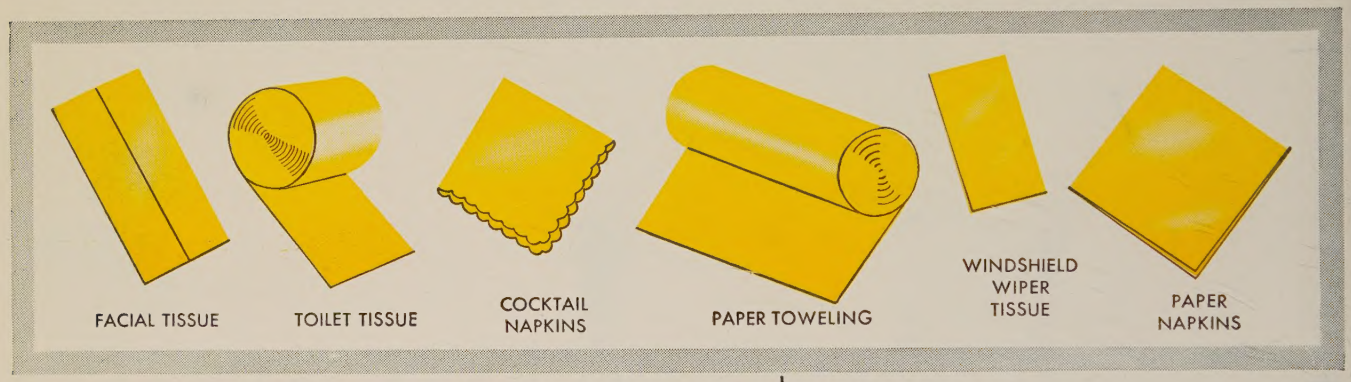

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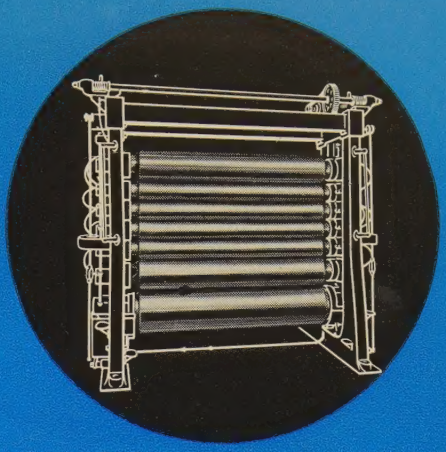

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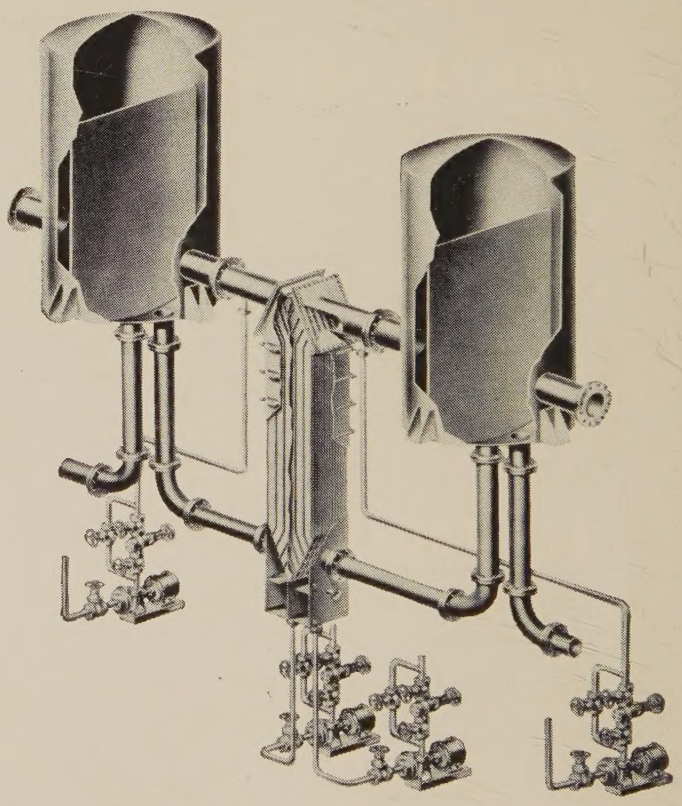

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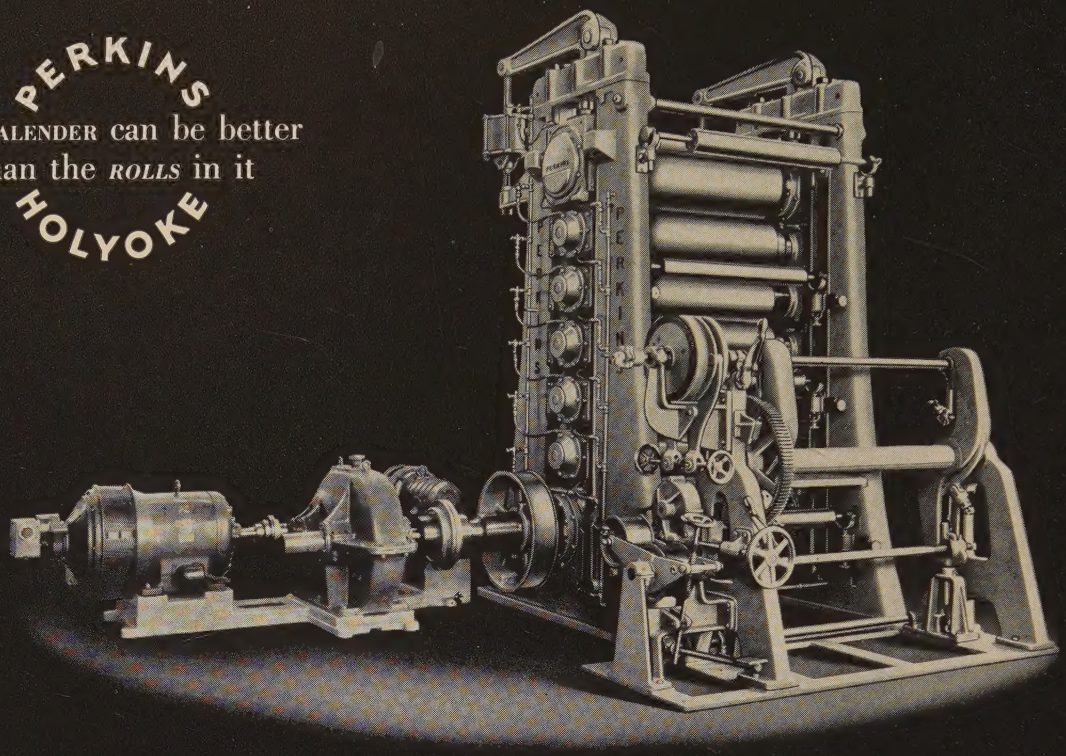

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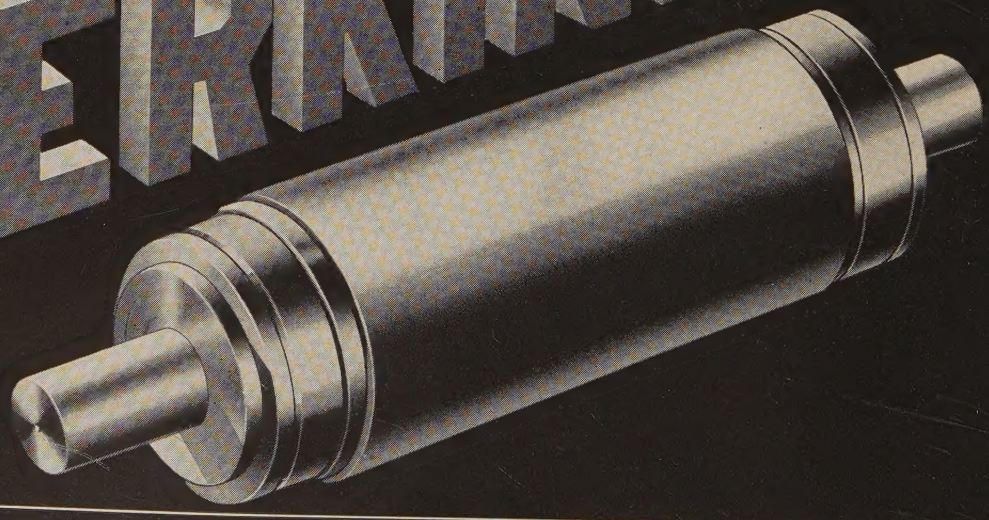

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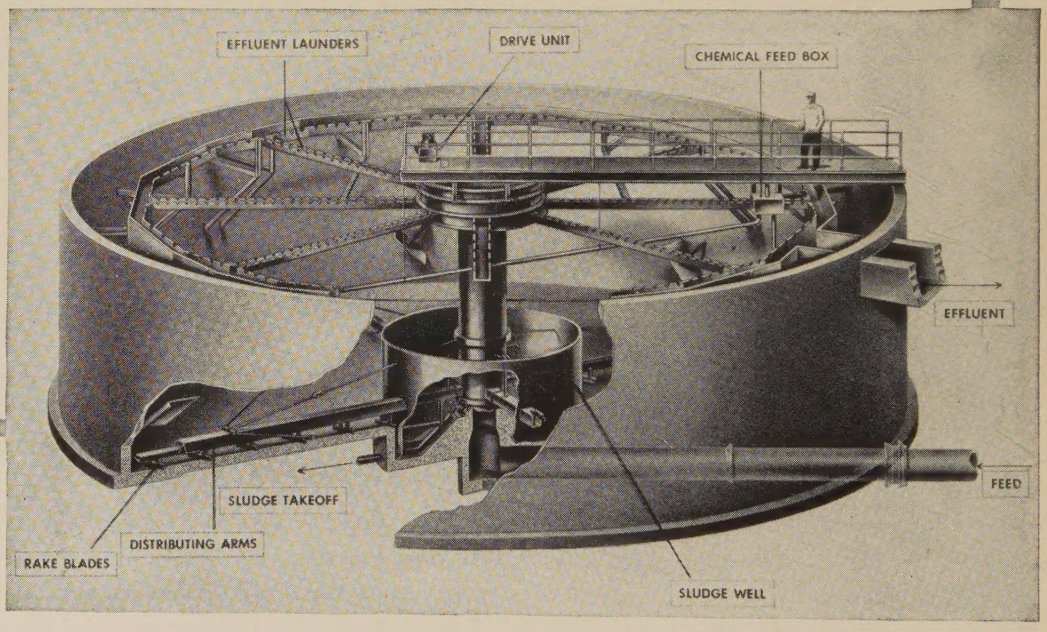

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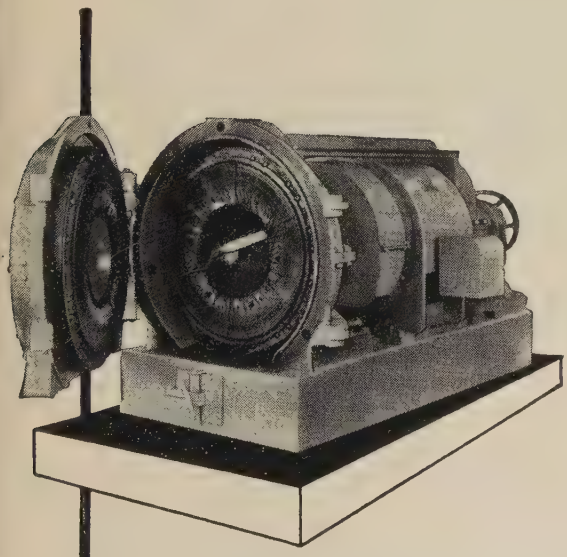

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T7-50

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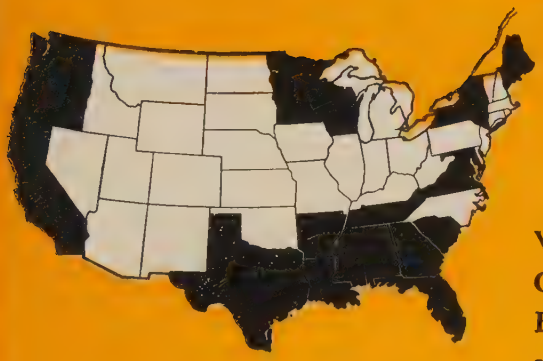
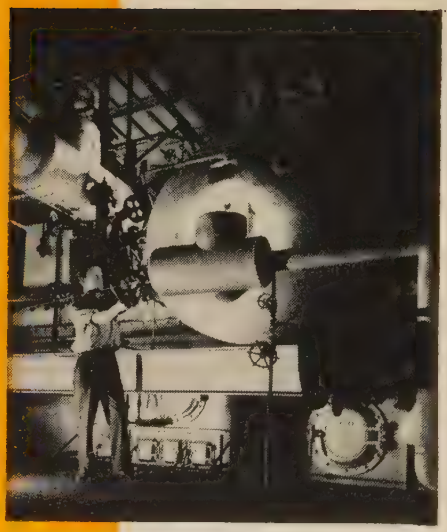


From Florida to Washington — from California to Maine — B&W Recovery Units serve the nation's vast wood-pulping industry.



Having a rated fuel capacity totaling almost 50 million pounds of dry solids per day, these B&W units efficiently recover heat and chemicals from waste liquors in kraft, soda, and magnesium bi-sulphite pulping processes. Since World War II, 31 units have been installed (or ordered), with an average steam capacity of 90,860 pounds per hour.

The performance of these units is proof that B&W has figured prominently as a pioneer in modern recovery techniques for the pulp and paper industry.

BABCOCK & WILCOX

P-53

Corrosion Problems in Sulphate Pulp Mills

CARL-GUSTAV VON ESSEN

During recent years the corrosion problems in sulphate pulp mills have grown more and more serious, and have been a subject of considerable discussion at the annual meetings of the Association of Swedish Paper and Cellulose Engineers.

The discussions at the winter meeting of 1948 resulted in a unanimous request from the Sulphate Section, to the Board of Directors, that a special committee should be appointed to study this question. The result was that the matter was submitted to the Sulphate Committee. At their meeting on April 23, 1949, a corrosion committee was appointed, consisting of R. Hellerqvist, Östrand, Chairman, L. Magnusson, Skutskär, and the author. As an adjunct expert of the iron and steel manufacturers, the committee had A. Franzen, Hagfors, a mining engineer. In addition the head of the Metallurgical Institute, Professor E. Rudberg, has been kind enough to help us with some advice.

Before any experiments were planned in detail, the members of the committee visited all the sulphate pulp mills in the country. The purpose of these visits was to make a survey of the corrosion troubles and gather advice. From his own experience, the author wants to affirm that the committee's expectations were thereby satisfied to the greatest degree.

A short résumé will be given of the experiences that were made available to the committee during the visits to the mills. However, it must be remembered that the committee has considered it their business to study only the special corrosion problems in the sulphate pulp mills. The more common corrosion problems, such as those resulting from ordinary rust, have not been treated. They are considered to be outside the scope of our work, since they are the subject of investigation by the Royal Swedish Academy of Engineering Sciences.

MILL CORROSION PROBLEMS

During the visits to the mills, we went through each department with the above-mentioned objects in mind.

The Digester Building

In the digester room (1–5 in Fig. 1), the corrosion problems varied greatly in the different mills. Concerning the digesters, it was only in connection with the rivets and riveted seams that any substantial agreement was found. Almost everywhere the joints were welded. In one mill the corrosion between the plates had been so severe in the overlapping parts that a hole could be made with a hammer. This digester had been in use for 19 years. Because the entirely welded digesters are becoming more common, those troubles (referable to joint corrosion) will probably disappear.

With regard to other aspects of the digesters, the problems were very different. At some mills there was no serious corrosion and at other mills, serious corrosion of the digester plate occurred. In most cases it was established that the corrosion was more severe in the bottom cone. However, there were mills where the corrosion was worse on other parts of the digester—e.g., localized on only one plate. At several mills the bottom cones had been replaced after about 20 years of use. However, in recent years, a notable increase in corrosion has been noticed. It appears differently in the various mills affected by corrosion. Thus, in some digesters a corrosion of about 2 mm. has been observed within a year in one mill and up to 3 mm. in another mill, whereas other digesters in the same mills showed very little corrosion. Usually it was found that the old Pintsch digesters have withstood corrosion better than digesters delivered later. At one mill a Pintsch digester has been in use since 1907. The diagram in Fig. 2 shows the magnitude of the corrosion in a digester which was installed in 1943. The solid vertical line shows the original thickness of the metal (23 mm. in the top dome and 26 mm. in the rest of the digester). The horizontal dotted lines give the mean values of experimental borings made after less than 5 years of use. In the diagram, the minimum thicknesses for different steam pressures are given [7, 8, and 9 atm. (gage)] as calculated by the Boiler Association. It was noticed that the corrosion was more pronounced in the bottom where it sometimes amounts to an average of 3.1 mm. a year. From the borings it appeared that in this digester, which is equipped with a circulation system, the corrosion was most severe opposite the liquor inlet in the bottom. A water gas-welded Pintsch digester had been used in parallel with this digester and showed, during the same time, a maximum corrosion of less than 0.2 mm. a year.

In the mills which employed upright digesters with liquor circulation there were some corrosion troubles in heat exchangers (2), pumps (3), and valves (5). The tubes in the heat exchangers are constructed either of carbon steel or of stainless steel with 13.3% chromium. Most of the operators have had the experience that the life of the carbon steel tubes is limited whereas the stainless steel tubes have given satisfactory service. The life of carbon steel tubes at one mill was 3 years, whereas stainless steel tubes have been used for 8 years and still show no sign of corrosion.

The liquor charging valves, the blow valves, and the relief valves are subjected to the most severe corrosion. However, if stainless materials are used, corrosion may be avoided. At one mill corrosion has been eliminated by the use of open hearth steel for the blow valve, the sealing (joint) being of monel metal.

The circulation pumps are also easily corroded; that is why they are made, in several places, entirely of stainless steel.

The Diffuser Building

In the diffuser building (7–10 in Fig. 1) the experience

Carl-Gustav Von Essen, Engineer, Stockholm, Sweden.

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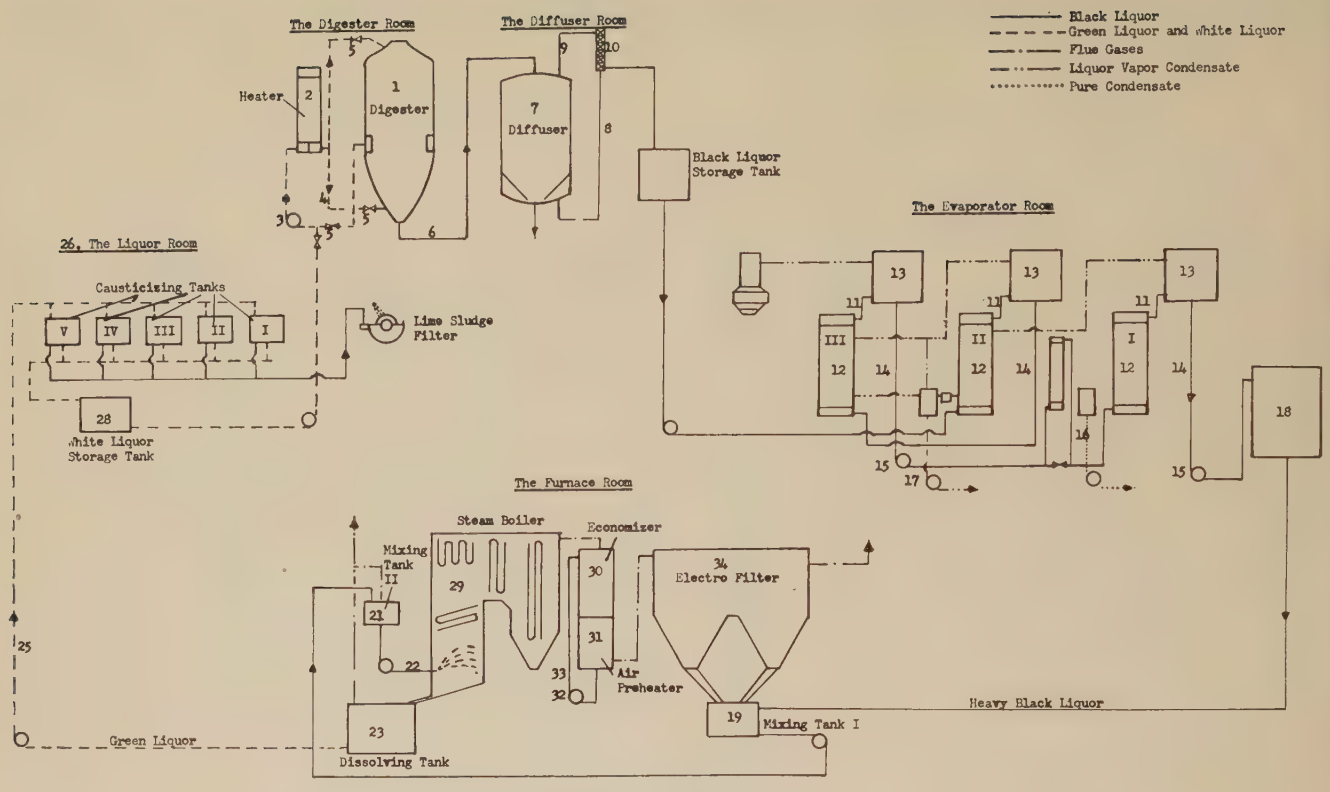


Fig. 1. Flow sheet for liquor, condensate, and flue gas sides in a sulphate pulp mill

of the mills seemed to be rather similar. The corrosion has mainly the character of pittings. They appear in the blowpipe lines (6), and in the lower parts of the diffusers (7). Thus, one mill reports that the life of a blowpipe line of steel had been only 2 years. At another mill it was mentioned that, after some years, the swing pipes had looked like they had been hit by birdshot.

In order to reduce corrosion in the diffusers, several mills have lined them inside, several times a year, with a concrete composed of 1 part cement and 2 parts sand.

The corrosion in the diffusers has forced several mills to replace the lower walls and the shell plates by new ones. At one mill they reported that this was done after 39 years' use of diffusers manufactured in 1902 (the corrosion was 9 mm.; the change in the thickness of the material was 11–2 mm.), after 17 years' use of diffusers manufactured in 1928 (the corrosion was 8 mm.; the change in thickness of the material was 14–6 mm.) and after 16 years' use of diffusers manufactured in 1932.

In a few mills they know from experience that the usual "red" steam pipes wear better than steel pipes in the pipe line between the central control of the diffusers and the diffusers, especially in the top lines (9). In the central control of the diffusers (10), corrosion was reported. At one mill the valves installed in 1946 have already become useless. Another mill having the same construction and the same type of valves has operated for twelve years without considerable corrosion.

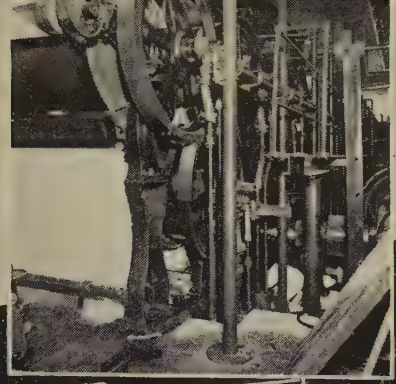
The Evaporators

The experiences of the mills regarding evaporators seem to agree (11–18 in Fig. 1) rather well, although the magnitude of the troubles may vary. Corrosion occurs both on the liquor and the liquor steam sides. It seems to have increased considerably in connection with the change to the new recovery systems in which the black liquor is evaporated to a rather high concentration and sprayed into the combustion chamber of the boiler. As an example, it may be mentioned that at one mill with injection (spray) apparatus the life of carbon steel pipes was only half a year, corrosion being very severe. At another mill with a similar recovery unit, the corrosion was of 14 kg. per sq. m. per year.

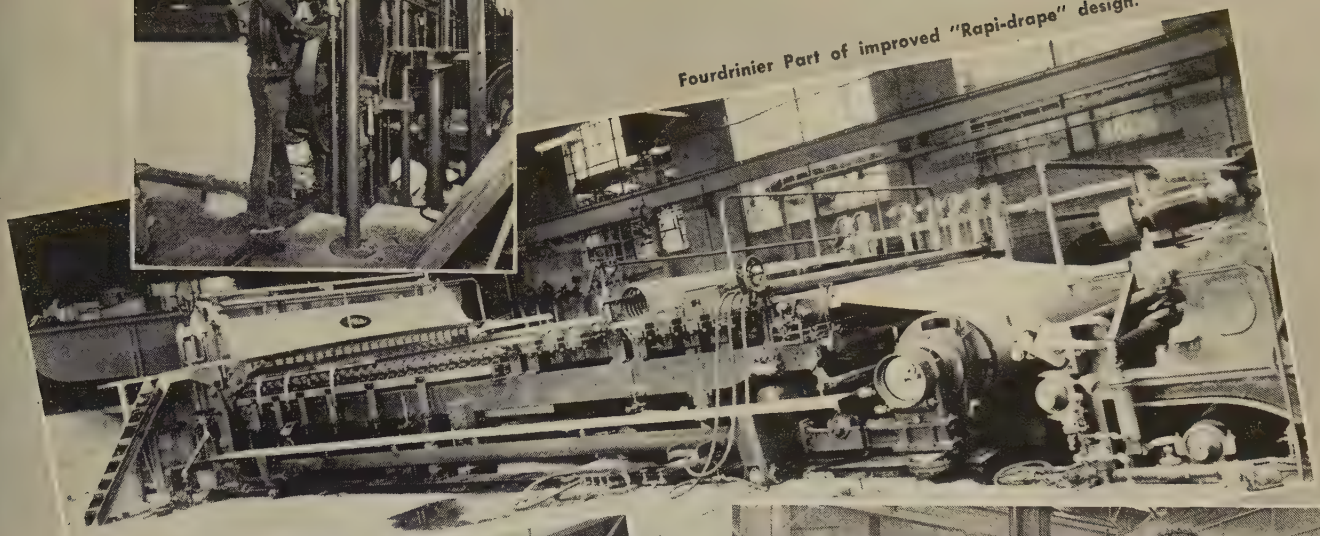
Generally, the increased corrosion appears to depend on the increased sulphidity and the higher concentration. In order to avoid this corrosion, some mills have begun to equip their plants, to a considerable degree, with stainless steel. However, other mills are afraid of this method because they believe that there is a risk of an increased corrosion caused by galvanic circuits.

Furnace Section

In the furnace room (19–24 and 29–34 in Fig. 1) corrosion has increased in proportion to the installation of the modern furnace units. In the black liquor system it is mainly the spray pumps which are more subject to corrosion. This is probably caused not only by the corrosion of the liquor, but also on mechanical erosion by the salt cake dispersed in the liquor. In the green liquor system the corrosion has increased considerably with the increased sulphide content of the liquor. In one mill, the life of the green liquor pump was reported to be only a few months. In the flue gas system, the economizer, the air preheater, and the electro filter are especially susceptible to corrosion. The trouble with the first two may be eliminated by the recently introduced sand cleaning method which will replace the usual method of water washing. The



Improved Steam Joint on dry part having open type gearing.



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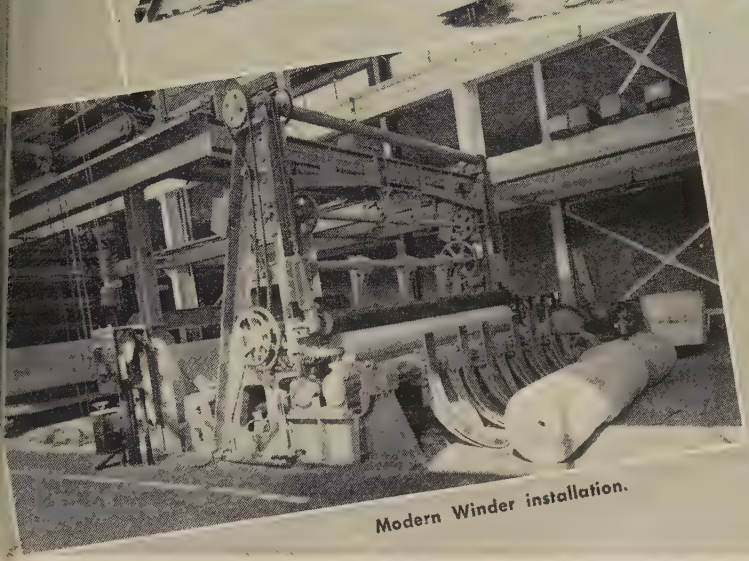
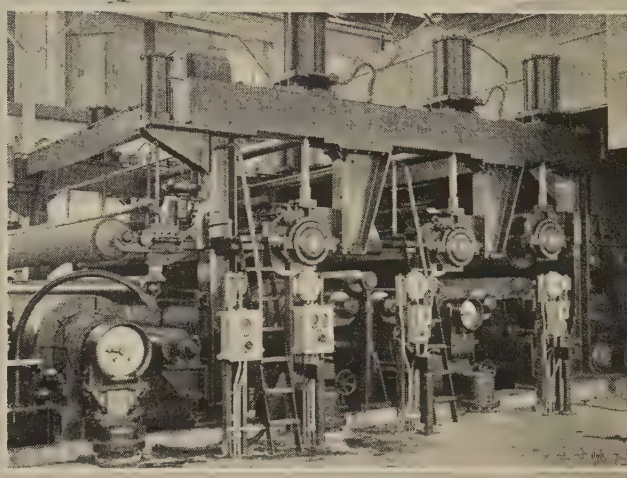


Modern Winder installation.



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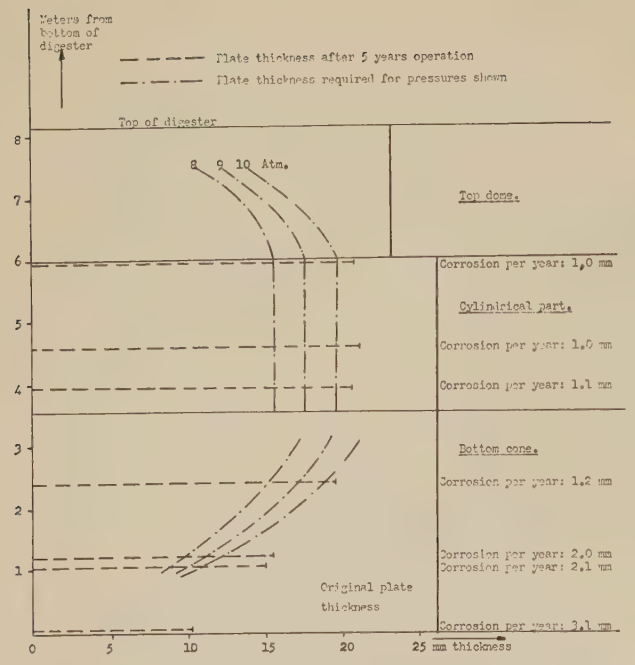
Meters from bottom of digester
Plate thickness after 5 years operation
Plate thickness required for pressures shown
Top of digester
8 9 10 Atm.
Top dome.
Corrosion per year: 1,0 mm
Cylindrical part.
Corrosion per year: 1,0 mm
Corrosion per year: 1,1 mm
Bottom cone.
Corrosion per year: 1,2 mm
Corrosion per year: 2,0 mm
Corrosion per year: 2,1 mm
Original plate thickness
Corrosion per year: 2,1 mm
mm thickness

Fig. 2. Corrosion in sulphate digester after 5-years' operation

most radical solution for the electro filter is to make the entire filter unit with concrete.

It is easy to realize that the disadvantages and advantages of the new recovery furnaces are not yet well known to mill operators. However, they already indicate severe corrosion to which more attention must be given.

Causticizing Room

Corrosion problems in causticizing (25–28 in Fig. 1) seem to have increased with the new recovery furnaces. In this case it is easily possible to avoid corrosion by the use of concrete. Unfortunately it is impossible to apply this protection everywhere. Therefore, increased difficulties are reported in regard to pumps and pipes if they are not made entirely of stainless steel.

RESEARCH PROJECT

After this survey was made, we were ready to plan the investigational work in detail.

The corrosion committee is very conscious of the complicated nature of the problems and of the truth of the proverb: "Aim at the stars and you may reach the treetops." Therefore, the committee proposed to obtain as complete answers as possible to the following questions:

1. What is the proper composition of materials for the sulphate pulp mills to withstand the attack of the chemicals used?
2. What are the possibilities for the protection of the existing materials against continued corrosion?
3. Is it possible, by a suitable treatment of the liquors and condensates, to reduce the corrosion?

The above survey indicates that there are two different types of corrosion. During the study of the abovementioned problems the conditions in the liquor systems of the mill (green liquor, white liquor, and black liquor) and in the condensate and flue gas systems must be considered separately. In order to handle corrosion problems the first important step must be to obtain definitive knowledge concerning the actual reason for, and the progress of, the corrosion.

Regarding the liquor system, the sulphate technicians seem to be of the same opinion—i.e., corrosion has increased rapidly with the higher sulphidity connected with the new recovery furnaces. This would infer that the corrosion is caused by the sodium sulphide in the liquors. In order to prove this, the following experiments have been performed at Delary: a sample plate from a digester was treated with hydrochloric acid and then washed clean with water. Immediately after weighing, the sample was placed in a glass beaker containing white liquor at a temperature above 40°C. The pretreatment was intended to destroy any possible oxide film. Under the chemical and heat conditions which prevail in the digester and in other places of the liquor system in a mill, this oxide film is supposed to disappear. However, a marked formation of iron sulphide on this specimen of plate could be observed immediately. It was so rapid that, within a few minutes, the white liquor in the beaker became quite black and opaque.

The same experiment was repeated with a sample of plate from the same digester which had not been pretreated with hydrochloric acid. As can be seen from the diagram in Fig. 3, the loss of weight of this sample was rather insignificant. No iron sulphide formation would be observed. After 13 days this specimen was moistened with a drop of hydrochloric acid and immediately washed with water. After this, the corrosion was marked as with the first specimen. Formation of iron sulphide was also observed.

These experiments gave valuable indications on how to perform corrosion tests on a laboratory scale. The pretreatment has been used also in the laboratory tests described below. The tests are considered proof of the fact that the corrosion in the liquor system generally is a result of the formation of iron sulphide. Thus, it can be related to the amount of sulphide in the liquors. Moreover, the conclusion may be drawn that the corrosion observed at many places in the bottom of the digesters may be related to the circulation speed of the

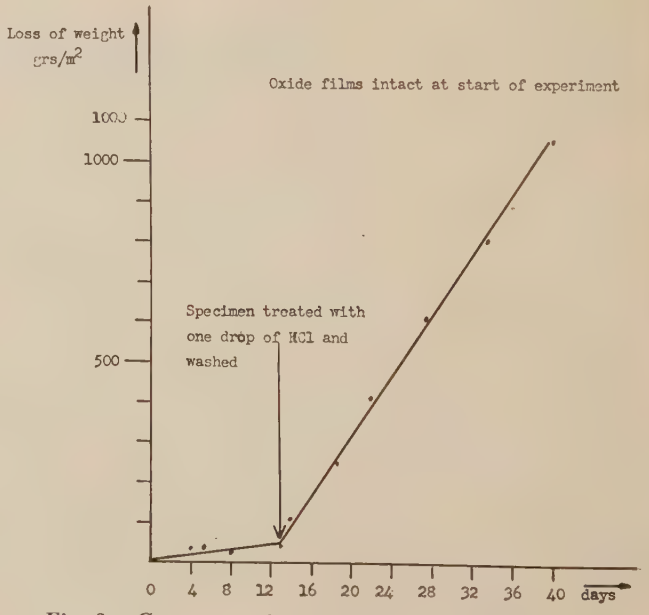
Loss of weight grs/m²
Oxide films intact at start of experiment
1000
500
Specimen treated with one drop of HCl and washed
0 4 8 12 16 20 24 28 32 36 40 days

Fig. 3. Corrosion of digester plate in white liquor

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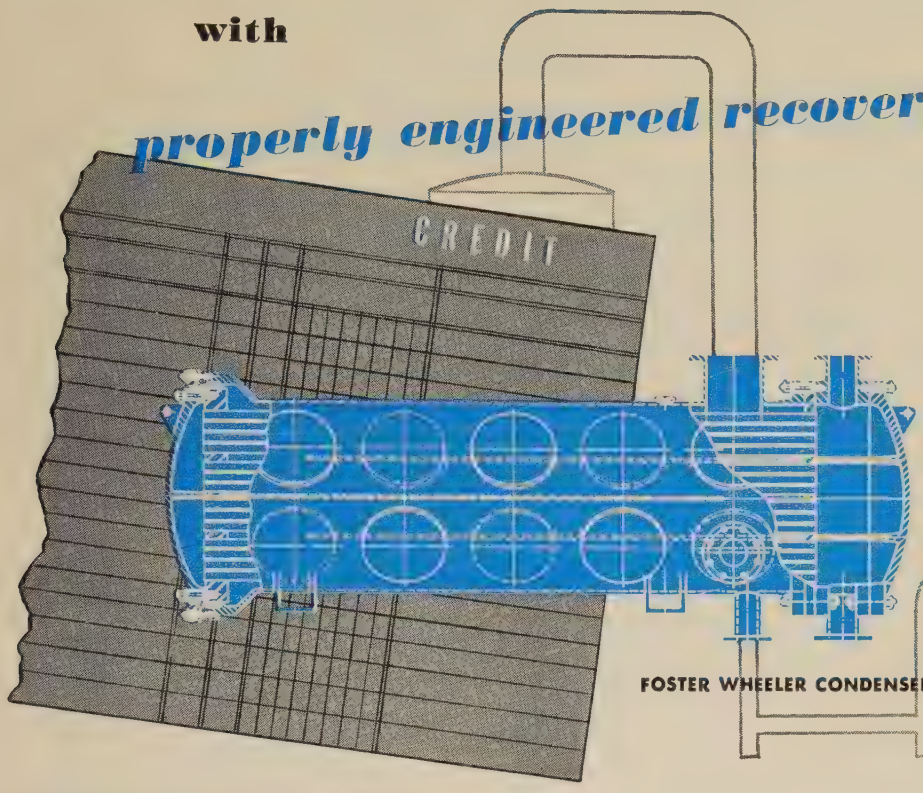

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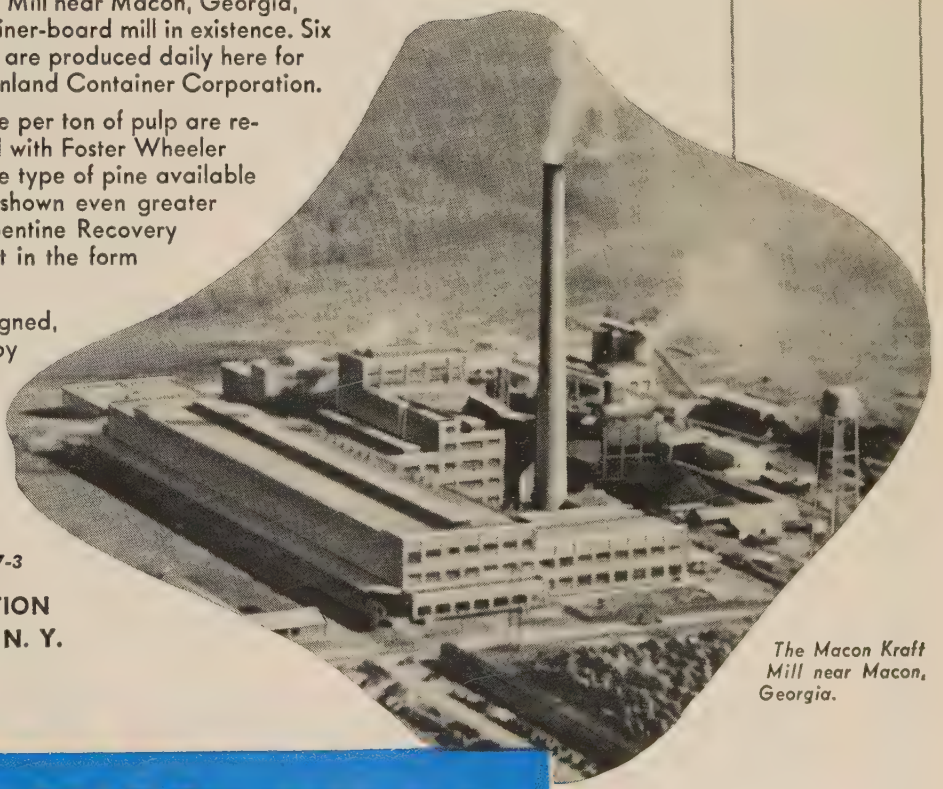

The Macon Kraft Mill near Macon, Georgia.

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Table I. Analysis of Liquors and Condensate from Different Mills

Mill	Black liquor before evaporation NaOH, g./l.	Black liquor before evaporation Na_2S, g./l.	Liquor vapor condensate n/10 J, ml./l.	Liquor vapor condensate pH	Liquor vapor condensate Solids, mg./l.	Liquor vapor condensate Ash, mg./l.	White liquor NaCl, g./l.
A	..	..	..	8.0	..	..	..
C	0.7	11.4	1.6	6.7	46.5	14	5.3
D	5.5	9.8	..	..	..	..	13.1
F	2.1	5.7	14.0	7.7	130	35	7.8
H	..	..	39.0	6.8	120	38	6.3
J	1.5	9.0	42.0	6.5	67	24	..
K	4.0	16.1	17.0	8.5	159	90	13.4
L	2.7	5.0	16.0	7.0	89	23	7.8
M	1.9	2.0	43.0	6.8	116	26	7.4
N	5.8	0.3	4.7	6.3	82	28	5.3
O	9.7	1.6	1.4	..	..	..	19.1
P	1.2	10.0	2.6	7.3	78	14	6.0
R	..	..	0	6.7	3.7	0	34.3

liquor. The greater the amount of cooking liquor passing through the rather narrow cross section at the bottom, the more favorable are the conditions for the formation of iron sulphide. This is in agreement with the data from the sulphate pulp mill at Sandviken, reported by Nykvist at the meeting last year. By decreasing the circulation speed, less corrosion was observed. Our investigations on the condensate and flue gas systems have thus far been limited to comparative analyses of the liquor vapor condensates. The analyses are given in Table I.

PROBLEMS IN THE LIQUOR SYSTEM

It seems to be fully evident that a change to stainless material can eliminate the corrosion problems. However, it is a rather expensive matter, even if in most parts of the equipment in the sulphate pulp mills, the material can be of the lowest quality—i.e., with about 13% chromium—and such material cannot be used everywhere. The change would, in many cases, be economical. Experience indicates that a cheaper solution might be obtained by the use of a more suitable composition of the usual plate material. These thoughts are primarily the result or the rather common opinion that the old Pintsch digesters have resisted corrosion better than those manufactured in more recent times.

In order to make these matters clear the Metallurgical Institute was asked for comparative analyses of plate material from a Pintsch digester and from a modern digester (manufactured in 1941). The tests included metallographic examination, determination of hardness, and chemical analyses. The metallographic investigation showed no difference. The grain size was estimated to be 12 for both specimens (measured with the J. K. M. scale). The hardness of the Pintsch digester plate was 131 Hb and of the modern digester plate 145 Hb. The chemical analysis gave the following result:

	Pintsch digester	Modern digester
C, %	0.11	0.11
Si, %	0.005	0.14
Mn, %	0.42	0.46
Cu, %	0.11	0.06
P, %	0.033	0.038
S, %	0.038	0.038

From this evidence the head of the Metallurgical Institute, Professor E. Rudberg, concluded that the modern digester is manufactured from case-hardened material and the Pintsch digester from material that has not been case hardened. The possible difference in corrosion resistance would then be related to the difference in the percentages of silicon and copper.

However, the old Pintsch digesters were usually of the rotary type, whereas the modern digesters are vertical and are equipped with circulation systems. This raises the question: is this difference in digestion method the cause of at least some of the dissimilarities? Plate material from both of the above mentioned digesters was available at Delary and some experiments were started there immediately. Specimens of plate from both digesters were placed on welded bolts opposite the liquor inlet in the bottom cone of a digester with a circulation system. A considerable loss in weight of these specimens could be observed after 2 weeks. The results are given below.

	Pintsch digester	Modern digester
Loss in weight after 12 days, %	1.6	3.8
Loss in g./sq. m. per cooking cycle	15.4	21.1
Loss in g./sq. m. per hour	2.71	3.20
Loss in weight after 120 days, %	17.4	39.6
Loss in g./sq. m. per cooking cycle	15.2	17.6
Loss in g./sq. m. per hour	2.69	3.8

These values agree rather well with the corrosion measured on digester plates in a regular cooking operation. It is about 3 mm. per year or 3.3 sq.m. per hour. As it appears in Fig. 4, similar results were obtained in the comparative laboratory tests described above.

Those tests show that there is a noticeable difference between the plate materials from the Pintsch digester and the modern digester. However, the difference is not as great as would be expected, nor does it seem to be a sufficient explanation for the difference in the observed corrosion. The conclusion may be drawn from these tests that the main reason for difference in the corrosion is to be found in the method of digestion. It is the sulphide content, the temperature, and the speed of the liquor which are most important.

In spite of this, we did not discard the idea of finding a proper composition of the plate without resorting to stainless steel. Because of the conclusions of Professor Rudberg, we decided to investigate the importance of the percentage of silicon.

Plate specimens were made with different percentages of silicon, manganese, sulphur, copper, and nickel. These specimens were tested in the laboratory, and in digesters with circulation systems at Delary; in two mills in the liquor system of the evaporation units and finally in two mills in the liquor-vapor condensate

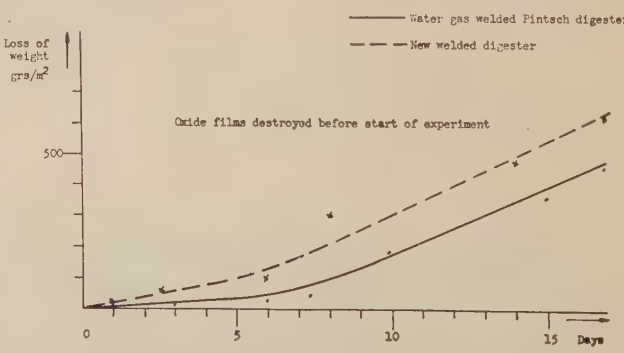

Fig. 4. Corrosion in white liquor of plate from Pintsch digester and digester made in 1941

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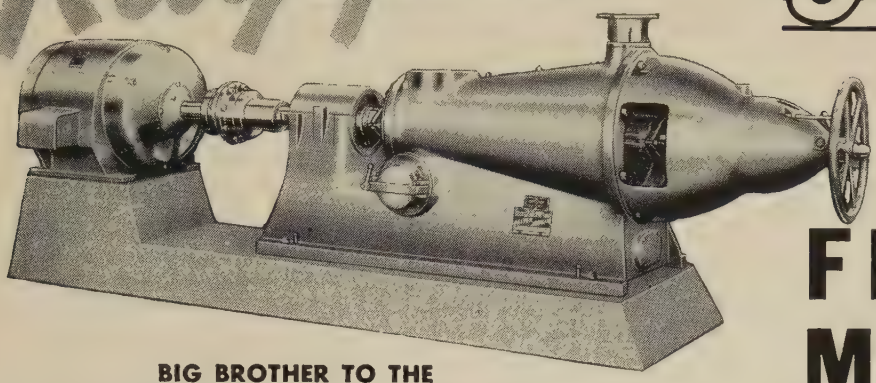

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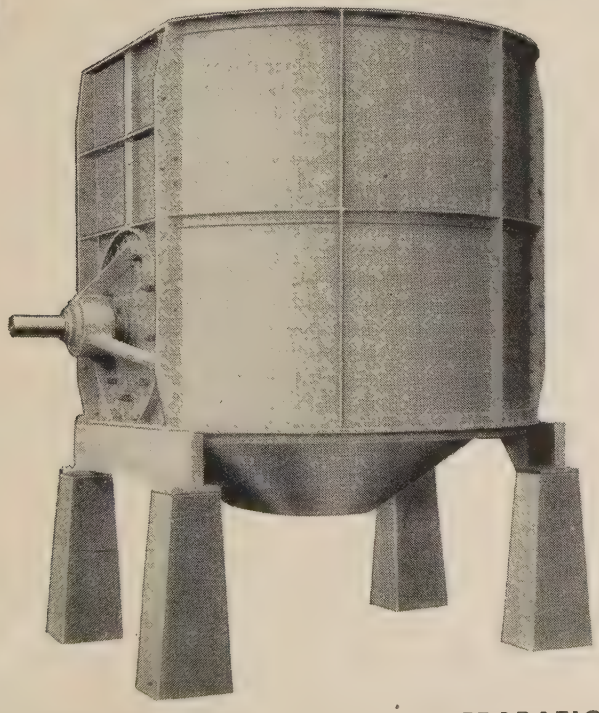

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Table II. Corrosion in Different Evaporator Rooms

Sample	SS 1[c]	SS 3[d]	R 2	R 4	R 7	R A[a]	D 2[b]
Analysis, %:							
C	0.10	0.06	0.16	0.15	0.15	0.15	0.10
Si	...	...	0.15	0.14	0.20	0.08	0.01
Mn	...	...	0.57	0.57	0.37	0.55	0.27
Cr	13.5	18	...	...	...	...	...
Ni	...	9	...	...	...	...	...
S	...	...	0.029	0.025	0.019	0.024	0.011
P	...	...	0.003	0.009	0.026	0.030	0.008
Cu	...	...	0.10	0.09	0.25	0.06	0.08
Corrosion on liquor side:							
Mill D g./sq. m./hr.[e]	0.004	0.005	0.20	0.23	0.19	0.26	0.21
Mill G g./sq. m./hr.	0	0	3.86	4.15	3.34	4.48	...
Corrosion in liquor vapor condensate:							
Mill B g./sq. m./hr.	0	0.006	0.93	0.87	0.82	0.76	0.96
Mill G g./sq. m./hr.	0	0	0.65	0.71	0.52	0.74	...

[a] Common steam pipe.
[b] Steel pipe.
[c] Stainless steel 1 (13.5% Cr).
[d] Stainless steel 3 (18% Cr, 8% Ni).
[e] Samples suspended in the bottom of the evaporator effect from which the thickest liquor comes.

system. It appears from the results in Table II, that the practical tests did not give a positive answer to our first question. Only the stainless qualities seemed to show good resistance.

The same result was obtained from the laboratory tests at Delary (Table III). However, these tests showed another matter of interest. They were started when the old recovery unit, equipped with disk evaporators and rotary furnaces, was still in use. They were continued during and after the start of a new recovery furnace with an injection unit, electro filter, and multiple evaporation. The tests gave a rather clear picture of the way in which corrosion by white liquor increases with the higher sulphidity obtained by the new recovery furnace. The data are given in Table III.

The variations in the composition of the different plate specimens were too small to be used as dependable evidence regarding the importance of the different alloy constituents. Only the tests on nickel in the digesters showed its beneficial influence. The addition of 5% nickel resulted in a considerably higher resistance. Unfortunately, the presence of nickel impairs the welding properties of the material. According to welding experts, materials with less than 2.5% Ni are easy to weld. At higher percentages, cracks may form in the welded seams. With 5% nickel materials with thicknesses below 5 mm. can probably be welded. Tests indicate that, after heating to about 200°C., it is also possible to weld thicker pieces.

A new series of tests were started with plate made by the Hagfors Järnverk steel works. The specimens were made with larger variations. Cobalt-alloyed plate was included in addition to those containing low percentages of nickel. The materials were tested in 60% black liquor in the evaporation units at Östrand in the circulation pipe line of a digester, in the liquor-vapor condensate system of the evaporation units at Skutskär and in the laboratory, in a heat exchanger in the digester building, in the electro filter, in the liquor system of the evaporation units, and partly also in the liquor-vapor condensate system at Delary. The results appear in Table IV.

In the liquor systems these tests gave very good evidence of the influence of silicon. Starting those tests with K_4 in the digester building at Delary, one may obtain a good idea of the life of different materials. The specimen K_4 was identical with the digester plate used in the earlier experiments and was taken from a new digester manufactured in 1941.

Manganese does not seem to have an appreciable influence on the resistance against white liquor. On the other hand silicon seems to have destructive action. An increase in the percentage of silicon to 0.99% reduces the already short life of the plate by half; the

Table III. Corrosion of Different Steels in Laboratory and in the Mill

Sample	35	48	44	66	67	81	5 Ni
Analysis, %:							
C	0.11	0.09	0.12	0.16	0.14	0.14	...
Si	0.24	0.03	Traces	Traces	Traces	Traces	...
Mn	0.22	0.16	0.44	0.42	0.45	0.36	...
S	...	...	0.033	0.043	0.041	0.039	...
P	...	...	0.005	0.012	0.016	0.012	...
Cu	...	...	0.15	0.16	0.18	0.20	...
Ni	...	...	...	...	...	...	5.0
I. Laboratory test in white liquor from old type recovery system Sulphide 26–29%.							
Corrosion, g./sq. m./hr.	0.733	0.640	0.711	0.741	0.616	0.733	0.063
II. Laboratory test in white liquor from new recovery system.							
(a) Sulphide 29–34%							
Corrosion, g./sq. m./hr.	0.850	0.687	0.810	0.875	1.120	0.860	0.092
Increase compared with test I	+16%	+7.3%	+14%	+8%	+82%	+17%	+46%
(b) Sulphide 35–41%							
Corrosion in g./sq. m./hr.	0.938	0.655	0.902	0.925	0.833	0.891	0.122
Increase compared with test I	+28%	+2.3%	+27%	+25%	+35%	+22%	+94%
III. Working test in digester with circulating system made at the same time as laboratory test I. Corrosion in g./sq. m./hr.	3.15	2.36	1.72	1.78	1.91	1.58	0.31

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Table IV. Corrosion Tests on Different Steel Plates

Sample	Analysis, % C	Si	Mn	Ni	Co	Cr	Cu	Tests made at Delary — Corrosion in g./sq. m./hr. At the laboratory	In a heater of the digester	In the diffuser	At the evaporator room in the heavy black liquor	In liquor vapor condensate	In the electro filter	Tests made at Skutskär — In the circulating pipe of the digester	In liquor vapor condensate	Tests made at Östrand — In heavy black liquor (60%)
Mn steel																
2880	0.09	0.17	1.10	..	..	..	..	0.929	2.23	..	0.022	..	0.47	0.205	0.352	1.8
2880—O	..	..	..	..	..	..	..	0.830	2.67	..	0.026	..	0.47	0.208	0.344	3.8
Si steel																
2879	0.09	0.99	0.17	..	..	..	..	0.952	4.43	..	0.181	0.558	0.49	0.569	0.274	3.5
2879—O	..	..	..	..	..	..	..	0.940	3.98	..	0.266	..	0.51	0.556	0.291	3.4
2881	0.12	3.92	0.28	..	..	..	..	0.798	7.13	..	0.605	..	0.50	0.947	0.320	5.0
2881—O	..	..	..	..	..	..	..	0.907	6.14	..	0.707	0.536	0.56	0.945	0.341	5.3
Cu steel																
2882	0.10	0.07	0.15	..	..	..	1.14	0.846	1.04	..	0.014	0.474	0.45	0.168	0.306	2.1
2882—O	..	..	..	..	..	..	..	0.855	0.88	..	0.014	..	0.46	..	0.328	1.4
Ni steel																
2891	0.10	0.07	0.12	0.40	..	..	..	0.818	1.06	..	0.022	..	0.49	0.130	0.282	2.2
2891—O	..	..	..	..	..	..	..	0.867	1.19	..	0.022	..	0.44	0.138	0.311	2.1
2892	0.10	0.09	0.15	1.06	..	..	..	0.834	0.97	..	0.019	..	0.42	0.161	0.268	2.9
2892—O	..	..	..	..	..	..	..	0.840	1.01	..	0.020	..	0.48	0.154	0.266	1.6
2894	0.11	0.07	0.17	2.09	..	..	..	0.566	0.15	..	0.015	0.296	0.38	0.131	0.296	1.8
2894—O	..	..	..	..	..	..	..	0.400	0.13	..	0.016	..	0.43	0.105	0.258	1.0
5 Ni	0.09	0.06	0.25	5.02	..	..	..	...	0.14	..	0.013	..	0.30	..	..	..
Co steel																
2895	0.11	0.07	0.19	..	0.46	..	..	0.370	1.81	..	0.022	..	0.43	..	0.302	2.8
2895—O	..	..	..	..	..	..	..	0.275	1.28	..	0.021	..	0.50	0.147	0.308	1.8
2896	0.11	0.20	0.23	..	0.84	..	..	0.447	1.22	..	0.021	..	0.37	0.136	0.284	1.8
2896—O	..	..	..	..	..	..	..	0.237	1.03	..	0.026	..	0.51	0.152	0.261	2.3
2900	0.11	0.49	0.51	..	2.28	..	..	0.182	0.86	..	0.026	0.294	0.37	0.165	0.226	2.4
2900—O	..	..	..	..	..	..	..	0.122	1.34	..	0.030	..	0.49	0.189	0.232	1.9
2901	0.11	0.37	0.46	..	4.84	..	..	0.105	0.17	..	0.023	..	0.41	0.082	0.253	0.7
2901—O	..	..	..	..	..	..	..	0.066	0.19	..	0.026	..	0.45	0.105	0.221	0.9
Bessemer steel																
2898	0.02	0.01	0.02	..	..	..	..	0.817	0.95	..	0.030	..	0.37	0.129	0.319	1.5
2898—O	..	..	..	..	..	..	..	0.848	0.61	..	0.027	..	0.46	..	0.342	1.5
2898—OB	..	..	..	..	..	..	..	0.847	0.56	..	0.047	..	0.38	0.182	0.344	1.1
Aluminium densified steel																
H 3002	0.09	0.09	0.08	..	..	..	..	0.895	1.30	..	0.027	..	0.43	0.178	0.314	1.3
Cr steel							Mo									
Uddco 1	0.15	0.29	0.38	..	..	5.40	0.00	..	0.74	..	..	..	0.55	0.318	..	..
Uddco 2	0.15	0.26	0.33	..	..	5.30	0.52	..	0.91	..	..	..	0.53	0.305	..	..
Uddco 3	0.05	0.26	0.32	..	..	3.11	0.50	..	1.30	..	..	..	0.51	0.293	..	..
Uddco 6	0.07	1.56	0.44	..	..	6.51	0.00	..	1.88	..	..	..	0.48	0.824	..	..
Normal digester steel																
K 4	0.11	0.14	0.46	..	..	..	..	..	2.16	0.24	0.044	0.564	0.45	..	..	..
K 4B	0.11	0.14	0.46	..	..	..	..	..	1.65	0.31	..	..	0.54	..	..	..
Stainless steel																
Stainless 1	..	..	..	..	..	13.5	..	..	0.003	..	0.006	..	0.17	..	..	..
Stainless 3	..	..	..	8.0	..	18.0	..	..	0.011	..	0.007	..	0.28	..	..	..
Aluminium	..	..	..	..	..	..	..	..	..	..	..	..	0.001	..	..	..

Samples marked O are not annealed. Those marked OB are not annealed nor pickled. All other samples are annealed except the aluminium. K 4 is taken from the same digester as the samples used in the other tests described in this article. Sample K4B is the same steel spotted with stainless steel.

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corrosion would increase to between 6 and 7 mm. a year. With 3.92% silicon the corrosion increased 3.5 times which would mean a corrosion of no less than about 11 mm. a year at the most exposed place in the digesters.

Nickel and cobalt seem to have about the same influence. The specimen with 2% cobalt is evidently not as good as the corresponding nickel material but this may be due to the higher silicon content. With 1% cobalt the life of the plate was increased about two times and at 2% to more than ten times the original life. Higher percentages give still better results. However, cobalt alloys may be too expensive and the use of nickel alloys is to a certain degree limited by the welding difficulties. For pipes, pump bodies, and cast parts of other kinds, one should be able to use materials with 5% nickel without any disadvantage. As already mentioned, materials thicker than 5 mm. should not be used unless special precautions are taken during welding.

These tests give an indication of the corrosion in different places in the mills, and of the corrosions in different mills.

The cost of nickel alloys, when compared with carbon steel, is not so high as to prevent its use, particularly because of its increased corrosion resistance. If the price of the pipes of nonalloy material is equal to 100, the same pipes with 1% nickel are said to cost 138, with 2% nickel 142, and with 5% nickel 166. The corresponding price of stainless steel with 13.5% chromium is 360 and with 18% chromium + 8% nickel is 560.

The material problem in the flue gas and liquor vapor condensate systems does not appear to be solved by the use of any of the materials tested (Fig. 7). However, the electro filter aluminum plate showed a remarkable resistance and seems to be the proper material to be used in this place. The qualification is that no condensate is formed.

PROTECTION OF EXISTING PLATE MATERIAL

When the second question was raised we had several surface coverings in mind. In fact, in February, 1948 experiments with spray coating and with stainless material were tried desperately in order to save a digester. This digester was installed in 1943 and the corrosion in the bottom cone since that time has been 3.1 mm. each year.

At several times we hesitated to use the more noble materials in contact with iron, because it might cause increased galvanic corrosion by the liquors. Therefore, one of the most important tasks was a study of this situation. This is important not only for surface coatings but also for all other uses of stainless steel in the sulphate pulp mills.

Since the stainless steels lie on the positive side of iron in the electromotive series, it would be quite natural if galvanic corrosion was obtained on the iron. From voltage measurements between digester plate and stainless plate in white liquor (Table V), it appeared that in this case the potential difference was considerably lower than in usual electrolytes. The galvanic currents were so slight that hardly any corrosion could be caused by them. On the other hand, voltage measurements between digester plate and sprayed stainless material gave quite different voltages and current intensities than were observed in the measurements with stainless plate. This is shown in Table V. It was also observed that the voltage and the current intensities were considerably higher with the sprayed material than with the same material in the form of wire. Judging from experiences in other fields, the risk of galvanic corrosion ought to be considerably greater with the use of the sprayed material than when using ordinary stainless plate.

Table V. Potential Measurements in White Liquor and Black Liquor

	Volt	*Ampere*
In White liquor:		
Stainless 18–8 plate	0.11	0.0009
Sprayed metal 1	0.25	0.11
Thread metal 1	0.01	
Sprayed metal 2	0.45	0.23
Sprayed metal 3	0.35	0.03
Thread metal 3	0.11	
Plate with 5% Ni	0.30	0.07
In Black liquor 15°Bè:		
Stainless 18–8 plate	0.12	0.0008
Sprayed metal 1	0.17	0.0042
Sprayed metal 2	0.20	0.020
In Black liquor about 35°Bè:		
Sprayed metal 1	0.21	0.0073

Oxide films on the digester plate were destroyed with HCl. In all tests the digester plate was negative and was measured against the materials given in the table.

In order to investigate these conditions, the following experiment was performed in the laboratory:

Two specimens were taken from the same digester plate as in earlier tests. The oxide film was destroyed with hydrochloric acid. One specimen was placed in a glass beaker containing white liquor and the other specimen was placed in contact with a plate of sprayed stainless steel (in a second beaker containing white liquor. The white liquor was renewed twice daily. In both tests the same rapid formation of iron sulphide was obtained as in earlier experiments. However, after three days, the formation of iron sulphide ceased entirely in the test in which the iron plate was in contact with the sprayed stainless plate. In the test with a single piece of iron, the corrosion continued at the same rate. After 19 days the sprayed stainless plate was removed, so that only the iron plate was in the white liquor. The formation of iron sulphide began after 18 additional days and the corrosion proceeded at the same rate as in the other test (Fig. 5).

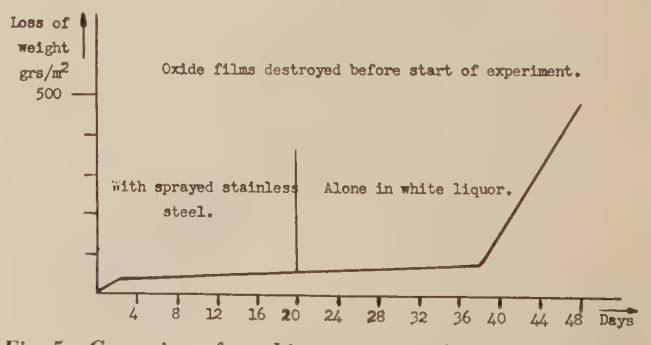
Loss of weight grs/m²
500
Oxide films destroyed before start of experiment.
With sprayed stainless steel.
Alone in white liquor.
4 8 12 16 20 24 28 32 36 40 44 48 Days

Fig. 5. Corrosion of steel in contact with sprayed stainless steel

This proved that in the galvanic couple formed by the digester plate and the sprayed stainless sheet iron there is a deposit formed on the digester plate which acts as an anode. The protecting deposit remains some

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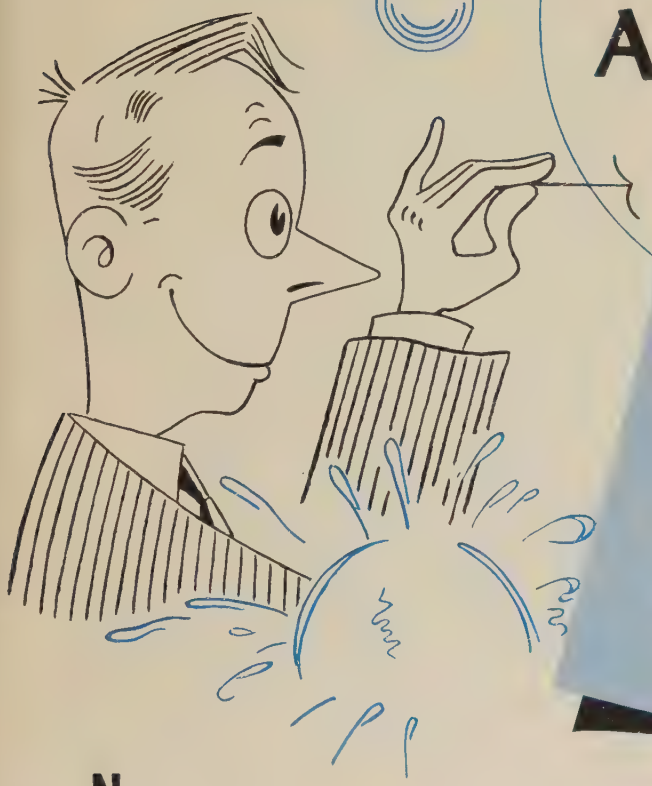

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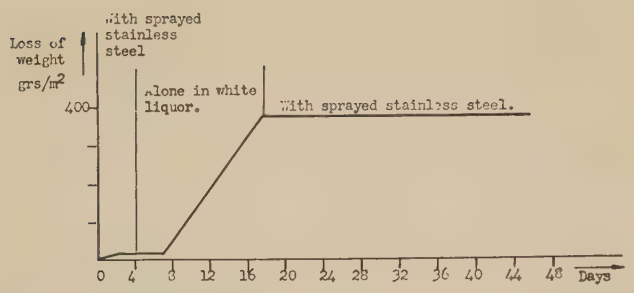

Loss of weight grs/m²
400
With sprayed stainless steel
Alone in white liquor.
With sprayed stainless steel.
0 4 8 12 16 20 24 28 32 36 40 44 48 Days

Fig. 6. Effect of galvanic currents on corrosion in white liquor

time after the circuit is disconnected. The experiment has been repeated several times with the same result (Fig. 6).

These tests show that, with the liquor used, there is no risk of galvanic corrosion in the liquor system. On the contrary, the results indicate that it is rather an advantage to form galvanic circuits in white liquor in which the plate to be protected is the negative pole. The greater the difference in voltage, the more advantageous the results.

This is exactly the reverse of the conditions in steam boilers and the like. In these cases one uses galvanic circuits for corrosion protection. However, one must select a material which lies on the negative side of iron in the electromotive series (usually zinc or aluminum). The Cumberland method is also contrary to the method indicated by the above experiments. In the Cumberland method the plate to be protected is connected to the negative pole of a direct current source.

In order to obtain additional evidence regarding the correctness of these conclusions, the plate specimen in white liquor was connected to the positive pole of a direct current source. A rod of the spray metal was connected to the negative pole. The white liquor was changed twice a day. During the first day a slight loss in weight was found which then ceased. The specimen had the same weight during the rest of the test (14 days). As would be expected, the same protection was obtained by a connection to a direct current source.

The experiments with spray covering started in February, 1948, confirmed these conclusions. The possibilities of metallizing, did not appear too promising because the coating came loose within a short time when used over large surfaces. We assumed that the insufficient adhesion was due to an imperfect cleaning previous to the coating. In fact, the whole work with the coating had to be done in the limited time which was available during a Sunday in order not to disturb the ordinary operations. Nevertheless, about 25% of the covering remained after 8 months. The test borings made by the Steam Boiler Association, showed that no corrosion had occurred in the bottom of the digester during this time. Even where the metal covering had become loosened after 1 week, the thickness of the plate remained unchanged. In another digester which was not spray covered on the bottom, the corrosion during the same time was 2 mm. or the same as that found earlier—i.e., 3 mm. a year.

The experiment with spray covering was repeated with another digester in June, 1948. A 700-mm. wide region was covered just under the drain plates in the cylindrical part of the digester. After the bottom cone had been replaced, this part was the weakest section of the digester. The cleaning was carefully done, which was evidently the reason that the covering adhered much better to the plate. This covering is still intact after 14 months. The improved strength is due in part to the decreased mechanical stresses.

A week after the spray covering had been installed, a thin deposit could be observed throughout the whole digester with exception of the sprayed part. It made the digester look moist. This deposit gave, after incineration, 27% Si, 21.7% Al_2O_3, and 32.2% CaO. The same deposit was obtained on test specimens suspended in the bottom of the digester.

There was also a deposit on the sprayed surface but it was of quite another character. It looked like the normal incrustations obtained in the circulation pipe lines and heat exchangers and, like them, consisted of calcium carbonate. This lime deposit gradually increased, as a result of which the galvanic circuit soon disappeared and the deposit impeded the favorable galvanic precipitation. After some weeks, holes could be observed in the deposit on the digester plate and after eight weeks the deposit had completely disappeared. The corrosion of the test specimens again reached the same value as earlier observed in the bottom cone.

In 1931, Professor Carl Bennedichs published in *Svensk Papperstidning* the results of numerous investigations over the nonuniform attacks on sulphate pulp digesters. He came to the conclusion that these attacks are not caused by any heterogenity in the plate

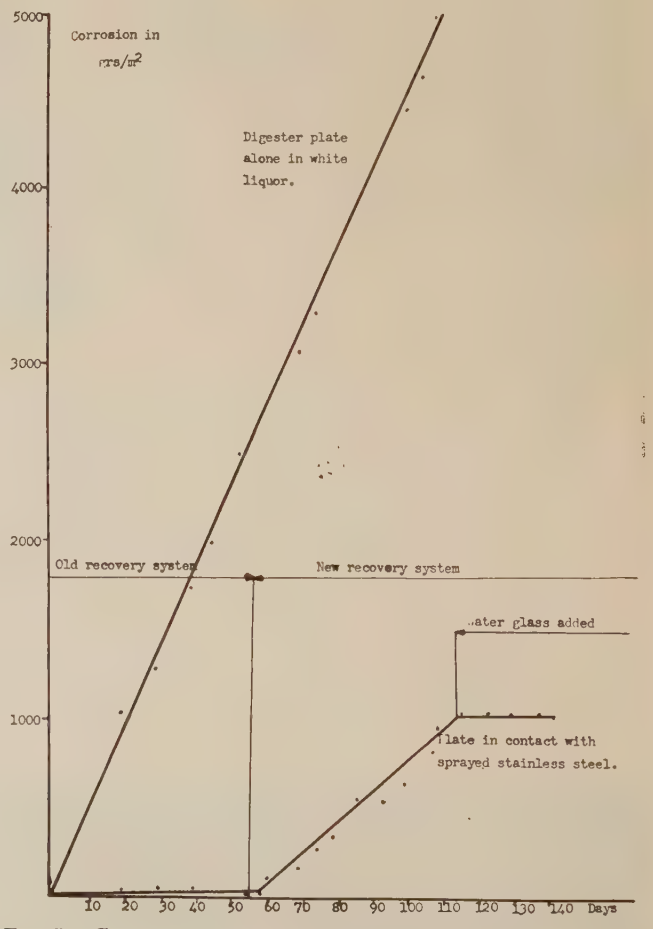
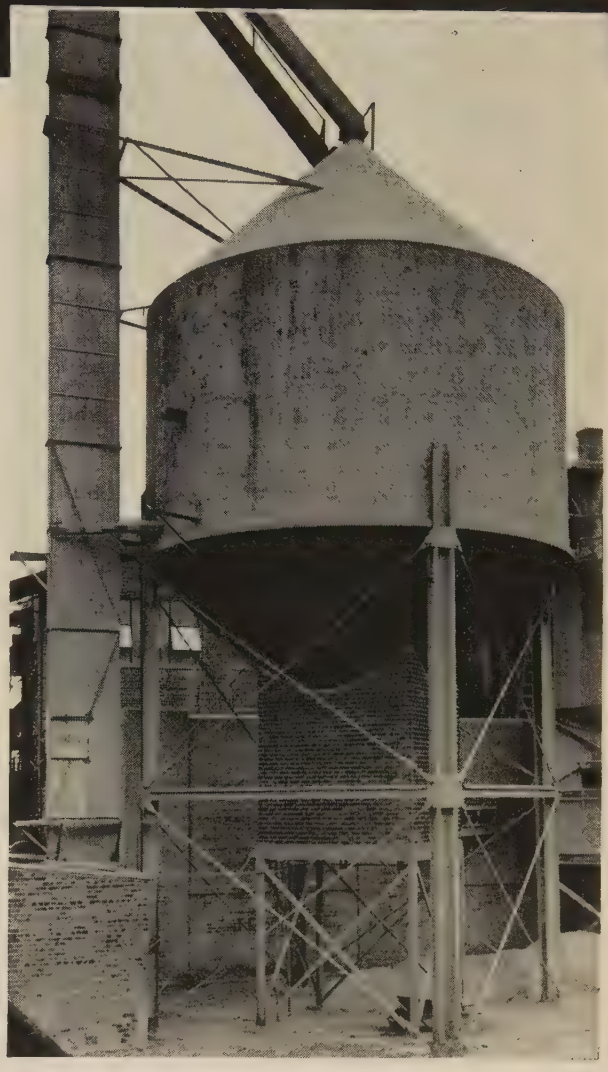


Fig. 7. Comparison of corrosion in white liquor from old and new recovery plants. (The tests are made in laboratory.)

Georgia mill uses Horton* structures in expanding capacity 100 per cent



The St. Mary's Kraft Corporation recently expanded the pulp producing capacity of its mill at St. Mary's, Georgia, from 250 to 500 tons per day—an increase of 100 per cent. The mill converts slash pine into pulp and manufactures kraft paper for shipping sacks, wrapping and other paper products.

Horton* steel plate structures were used extensively in this expansion program. We furnished eight digesters, several liquor storage tanks, a 15,000-bbl. fuel oil tank, a chip tank, a blow tank, a salt cake tank and a lime storage tank.

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Two of the Horton structures installed in connection with the expansion of the St. Mary's Kraft Corporation mill at St. Mary's, Georgia, are shown above. They are (left) 28-ft. diam. by 16-ft. salt cake storage tank and (right) 12-ft. diam. by 10-ft. lime storage tank*

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Table VI. Corrosion in White Liquor from Different Mills

Mill	Analysis of white liquor Na_2CO_3, g./l.	$NaOH$, g./l.	Na_2S, g./l.	Corrosion of digester plate g./sq. m./hr.,	Corrosion of the same plate spotted with stainless steel g./sq. m./hr. 1st–3rd day	4th–10th day
A Old recovery system	22.3	100.0	39.8	0.43	0.047	0.039
B Old recovery system	34.9	108.5	34.9	5.87	4.86	0.023
C New recovery system JMW	44.4	102.2	50.0	1.96	1.90	0.054
D New recovery system JMW	23.6	85.1	43.5	3.59	3.50	0.00
E New recovery system Tomlinson	37.7	114.6	39.7	4.10	5.10	2.99
F New recovery system Tomlinson	22.1	85.0	37.9	1.94	2.62	1.96

material. He thought that the plate is normally protected by a coating which, when broken off or washed away, exposed surfaces which are then attacked by the liquor.

The above-mentioned experiments in Delary complete these investigations. They indicate the conditions of the formation of the protective coating which Professor Bennedichs considered to be of decisive importance for the resistance of the plate to corrosion by the liquor. Moreover, they present a possible explanation for the different kinds of corrosions which occur in the different mills or in the same mill.

On the basis of the test results, it can be stated that it is possible to obtain a protection of existing plate material against corrosion on the liquor side, by means of galvanic currents, or by connection to a current source. An explanation of this might be that the silicic acid in the liquors is deposited on the anode and protects it against further attack.

We now asked the question: will the decreased content of silicic acid in white liquor, which follows from the new recovery furnaces and the spray unit, reduce the possibilities of galvanic protection? It appears from Fig. 7 that this seems to be the fact.

It is evident from the figure that an addition of silicic acid reduces the corrosion in the galvanic circuit to the same extent as in the liquor from the old recovery furnace. The silicic acid content of the liquor need not depend only on the conditions in the recovery system, but may also be influenced by the content of silicic acid in the lime and wood. These questions will be investigated further, as well as the importance of other constituents in the liquor. It may be stated that the liquor must contain a sufficient percentage of silica or other compounds, acting as anodic inhibitors, if complete protection is to be obtained by the galvanic method.

As a first step in our investigations of the composition of the white liquor for corrosion, the digester plate used in the earlier experiments was tested in the white liquors at different mills. At the same time tests have been carried out on the same plate, spot coated with a more resistant material in order to discover the possibilities of galvanic protection in different liquors. According to Table VI, the tests have confirmed the above conclusions. At the mills which have not yet changed to modern recovery furnaces, and at some of the mills with spray units, the corrosion could be stopped by galvanic means in the laboratory.

The tests with spraying and spot covering in the mill work have been continued at Delary. It appeared that the spraying and the spot coating have certain operating disadvantages which makes it difficult to obtain a lasting protection.

First, the spray-coated surface is very porous. Because of this, the precipitated calcium carbonate adheres easily to the surface. Also on the spot-covered surface the calcium carbonate is precipitated on the "noble" points and the galvanic circuit disappears rather soon.

Secondly, there are great difficulties in making spray coatings adhere satisfactorily to the base. Good adhesion is obtained more easily if the surface to be covered is rough. This is unfortunately not the case in digesters and the other vessels which one wants to protect in the sulphate mills. Therefore, it may be difficult to achieve a sufficient and durable protection by this method at the places where the mechanical stresses are great—e.g., in the bottom of the digester and other places. However, the drain plates in the digesters, the heat exchangers, and the evaporation units may be protected by spray coating. The same thing is true for the machine parts which can easily be provided with a rough surface to which the covering will adhere well.

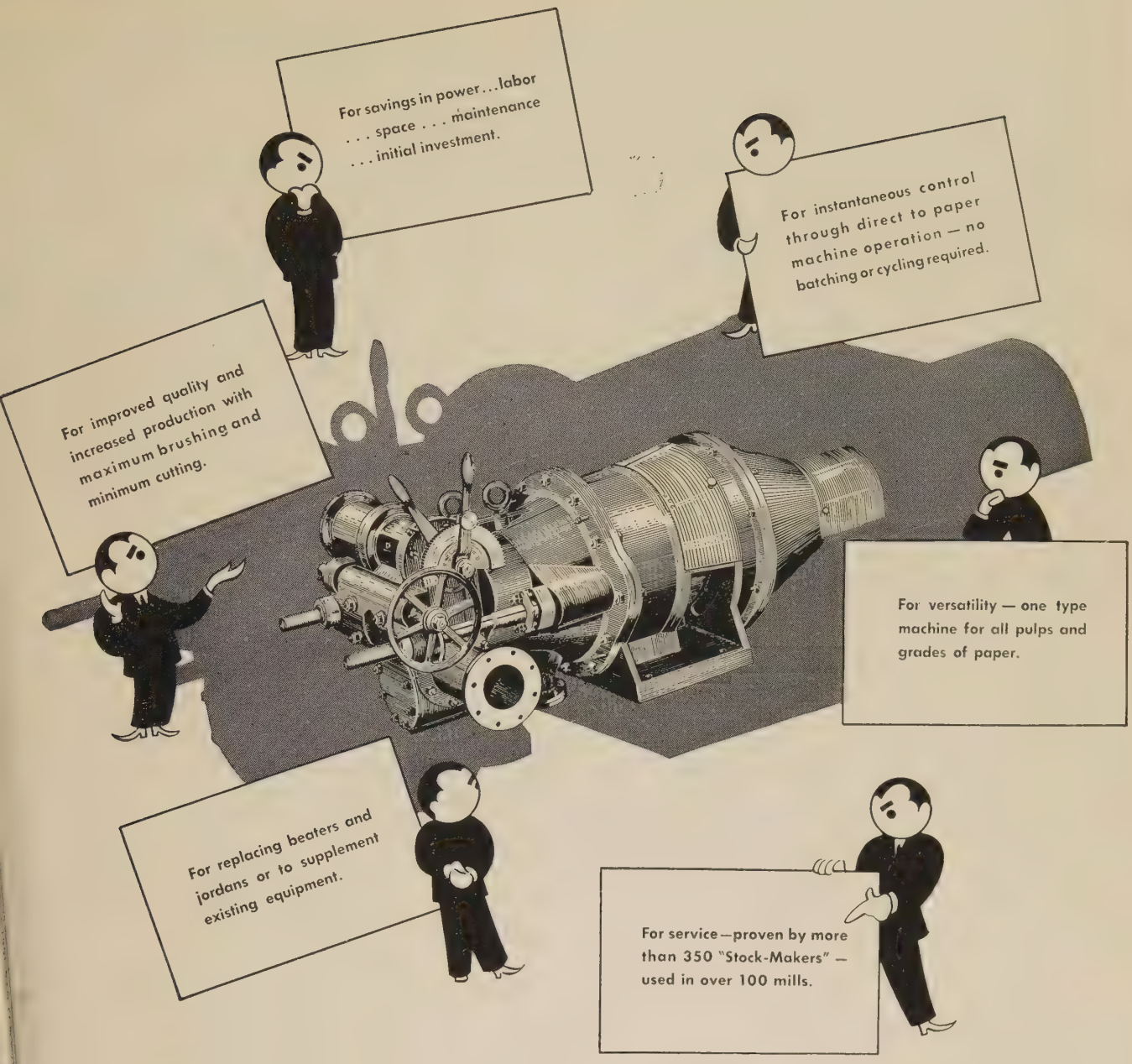
With spot coating (fusebound) it may be possible to obtain good adhesion for the spray coating but it will be a very expensive method to protect large surfaces. However, it will undoubtedly be possible to obtain good protection for smaller surfaces. In any case, it will be much cheaper than to cover plates which are severely corroded by welding. The latter method is used in some Swedish mills.

Tests were also run to investigate an economical method of connecting the parts to be protected to a direct current source.

Welding of stainless steel plate onto the exposed areas will not give the same strong galvanic effect as spray coating, but it has the advantage that the lime deposit does not adhere as firmly. In this manner one mill has been able to protect a digester against continued attack. The corrosion had been very bad at the bottom. The bottom cone, therefore, was lined with welded stainless steel plate. It appeared later that the corrosion in the other parts of the digester was also diminished. The explanation is given by the above mentioned experiments at Delary. With the use of stainless steel plate the joint to the plate, which is to be protected, must be so strong that the liquor cannot penetrate between the plates, otherwise, slot corrosion may occur with the same unpleasant surprise that most owners of riveted digesters have encountered.

The results of the experiments open wider perspectives. One can, of course, with good reason speculate on the manufacture of new apparatus of different materials for the liquor system. The most exposed parts should be made of more resistant materials. The possibilities are limited, however, because the coefficients of thermal expansion of the two materials must not deviate appreciably from each other. In any case, one need not hesitate to use materials with a larger resistance against corrosion at the exposed places in the liquor system of sulphate pulp mills.

The investigations of the flue gas and condensate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systems have not been concluded. We may expect galvanic corrosion from the condensate side by the use of, e.g., stainless steel tubes in the evaporation units or in the digester.

Many years of experience of several mills do not indicate any difficulties in this respect. On the contrary, they seem to have observed less corrosion and they have found some advantages which would make the use of stainless steel tubes advisable. Thus one obtains with stainless steel tubes an improved heat transfer rate in the evaporation units which use liquor vapor. Certainly, the heat transfer coefficient is better with new carbon steel tubes than with stainless steel tubes. However, the carbon steel tubes are attacked by the liquor vapor and deposits are soon formed on the tubes. These deposits decrease the heat transfer coefficients considerably and cause great difficulties in cleaning. On the other hand, the stainless tubes remain shiny.

At Delary we tried to measure the heat transfer coefficient for stainless steel tubes and carbon steel tubes after use for some months. Under the same working conditions there was obtained for carbon steel tubes a *K*-value of about 500 and for stainless steel tubes a *K*-value of about 900. This considerable improvement, which evidently is possible, must be considered in economical calculations of the potentiality to exchange carbon steel tubes for stainless ones.

It would also be reasonable to plate one side of the carbon steel tubes with nickel or chromium. According to the literature in this field, good results have been obtained in the United States by this method. Experiments at Skutskär showed, unfortunately, that the nickel plating is not resistant to corrosion in the liquor vapor system. With present prices chromium plating is considerably more expensive than stainless steel tubes. The behavior of nickel plating in the liquor system has not been studied. It should be possible by this method to procure a proper protection of the effects in which the liquor steam condensate is absent, which is the motivating force in the use of stainless steel tubes.

REDUCTION OF CORROSIVE EFFECT OF LIQUORS AND CONDENSATES

At the sulphate mill of Norrsundet we have obtained information about the effect of the oxidation of the liquor on the corrosion of black liquor. This was the origin of the first efforts of the corrosion committee in order to get an answer to our third question. Before the oxidation of the liquor by the Bergström-Trobeck method, the life of the most exposed carbon steel tubes in the evaporation units was 2 years and 7 months. Since oxidation of the liquor has been practised, carbon steel tubes have been used for a period of 7 years and still have not been changed.

This result has been verified by experiments at the sulphate mill of Östrand. The experiments were performed in the circulating black liquor of a film evaporator at 125°C. and 58% dry content. They show that the corrosion in the tubes is reduced from 14 to 2 kg. per sq. m. a year when the sulphur content in the incoming liquor (20 to 30% dry content) is reduced by oxidation to less than 2.5 gram Na_2O per liter. The heat transfer coefficient was not changed by the oxidation, nor could any changes be found in the viscosity or the elementary analysis of the black liquor.

Thus far the experiments show that the corrosion in the liquor system can be reduced considerably by oxidation of the black liquor. It is believed that oxidation of the liquor will decrease corrosion in the condensate system.

In the white liquor and the green liquor systems, however, corrosion will probably be increased by oxidation of the black liquor. In fact, this is followed by an increase in the sulphur content which, as regards the quality of the pulp, is an advantage. The oxidation of green liquor or white liquor can never be considered because it lowers the content of the desired sulphide. In order to protect against corrosion in this phase, the most effective method until now seems to be the selection of the proper material, and protection with the help of the galvanic currents related above.

However, we should not overlook the possibilities of reducing the corrosive effect of the liquors by using the proper composition. The marked difference in the corrosion of concentrated black liquor, measured at Östrand (14 kg. per sq. m. per year at a dry content of 58%) and at Delary (from Table IV, 0.8 kg. per sq. m. per year at a dry content of 60 to 62%), and the tests made on the liquors of different mills, all indicate dissimilarities in the composition of the liquors from different mills, which cannot be determined from the ordinary control analyses. Differences in the heat conditions and in the speeds of the liquors may be the origin of varying corrosion troubles at the different mills. It would be valuable to investigate the importance of the liquor composition, the speed of the liquor, and the temperature. As a first step in this direction the committee has requested all the sulphate pulp mills in the country to analyse their liquors and condensates accord ing to uniform methods. The results appear in Table I.

CONCLUSIONS

The work of the committee has led to the following conclusions:

1. The corrosion in the liquor systems of sulphate pulp mills depends mainly on the formation of iron sulphide and is caused by sulphide in the liquors.

2. Silicon makes the iron less resistant to corrosion in the liquor system. Even a small content of nickel improves the resistance.

3. There is no danger of galvanic corrosion in the liquor system with the investigated liquors, as long as the liquor is circulating.

4. Existing plate materials can be protected by the help of galvanic currents or connection to direct current. The amount of silicic acid and other inhibitors in the liquors, however, is important. On most exposed areas, i.e., where the concentration, the speed and the temperature of the liquor have maximal values, this protection is best obtained by the insertion of sheets or tubes of stainless material or by spray coating with similar materials.

5. The heat transfer can be improved by the use of stainless steel tubes in the evaporators effects which are heated by liquor vapor.

6. Tests in the liquors from different mills indicate that an appropriate composition of the liquors may reduce existing corrosion. These experiments are not yet concluded.

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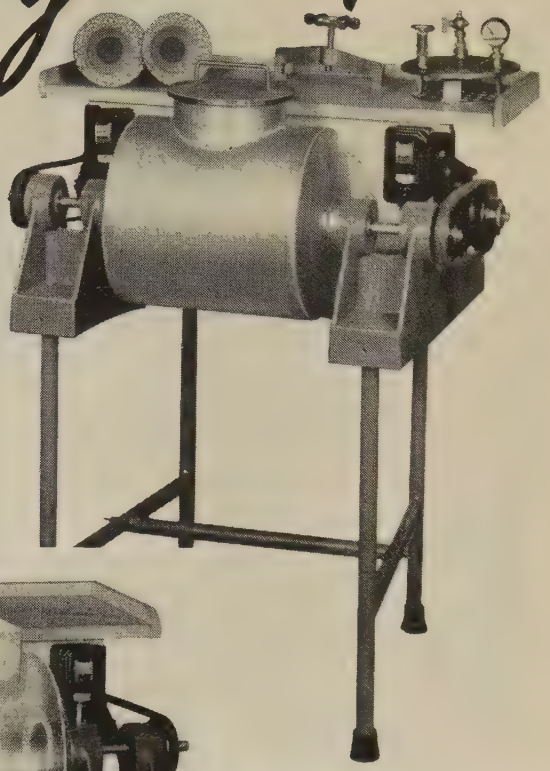

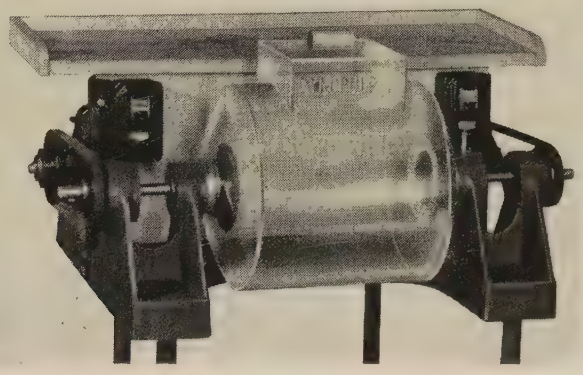

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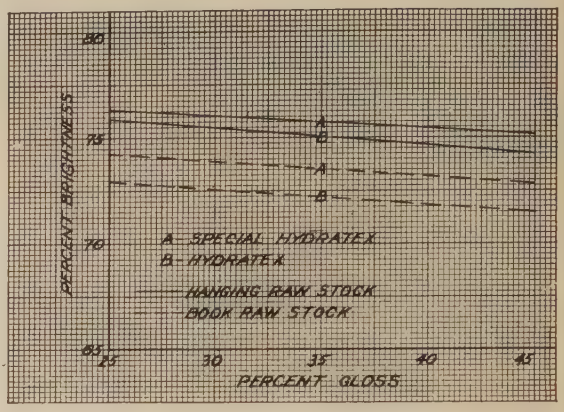
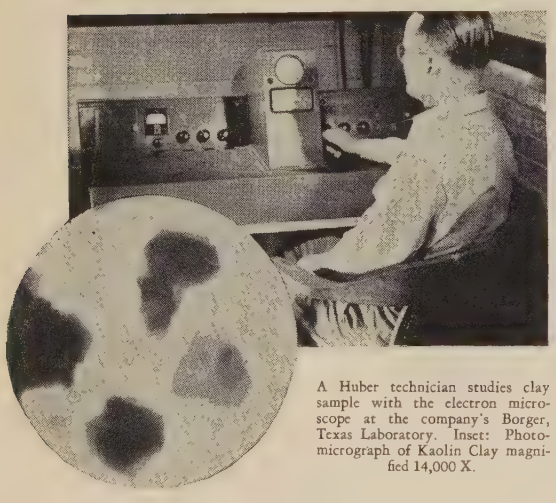
NUMBER 4 OF A SERIES FROM THE J. M. HUBER CORPORATION LABORATORIES

TESTS SHOW HOW CALENDERING REDUCES BRIGHTNESS OF COATED PAPER SURFACES. An uncalendered coating surface possesses the lowest gloss and the highest brightness. Calendering reduces the brightness but enhances the polish or gloss of the surface. In the Huber Laboratory surfaces treated with *Hydratex* and *Special Hydratex* clays were calendered to give different degrees of gloss as determined on the Bausch and Lomb glossmeter, and then tested for brightness with the General Electric Reflection Meter. The accompanying graph shows how brightness decreases with increased gloss. Both *Hydratex* and *Special Hydratex* are Huber coating Clays. In *Hydratex*, 70–72 percent of the particles are less than 2 microns in diameter; in *Special Hydratex*, 78–81 per cent.



Effect of Clay Fineness on loss of brightness due to calendering in a starch-clay coating



A Huber technician studies clay sample with the electron microscope at the company's Borger, Texas Laboratory. Inset: Photomicrograph of Kaolin Clay magnified 14,000 X.

QUALITIES OF A SUCCESSFUL COATING PREPARATION. A coating preparation must fulfill a number of requirements. It must penetrate and bond satisfactorily to the surface of the paper. It must flow sufficiently well to permit the required degree of spreading at the machine speed, and it must have the property of leveling and setting after being applied to the surface of the paper. It should be of such a character that on drying, a smooth, even surface is produced which can be calendered to produce gloss without the application of undue pressures. While most of these properties are influenced by the nature of the adhesive material, the various pigments have a considerable effect. Kaolin clays have long been established as excellent pigments to satisfy the requirements of a successful coating preparation.

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HOW WATER WASHED AND AIR-FLOATED FILLER CLAYS COMPARE. The chief advantage of washed clay is its less dusty nature. It also has about twice the bulk density of normal air-floated clay, and can be handled without a pneumatic conveying system. Smaller mills may take washed filler in bulk and unload it manually, or by belt, bucket, or screw type conveyor. Huber's "Aerfloted" clay, however, with its low moisture content (1–2%, compared to 4–7%) can reduce freight costs up to 5%. "Aerfloted" clays, too, are less expensive and equal or surpass water washed fillers in quality. Leading paper mills have standardized on air-floated filler.

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Above 5	14.6%	27.9%	14.6%	28.0%
4–5	5.0	5.2	4.7	8.1
3–4	6.7	6.7	6.8	9.8
2–3	9.9	8.7	9.9	15.6
1–2	15.8	12.5	15.5	16.0
0.5–1	18.0	11.3	17.1	16.0
0–0.5	30.0	27.7	31.4	6.5
Above 5	14.6	27.9	14.6	28.0
Under 2	63.8	51.5	64.0	38.5

"Aerfloted" Clays are automatically weighed during bagging. They have an apparent density of 25–30 pounds per cubic foot.

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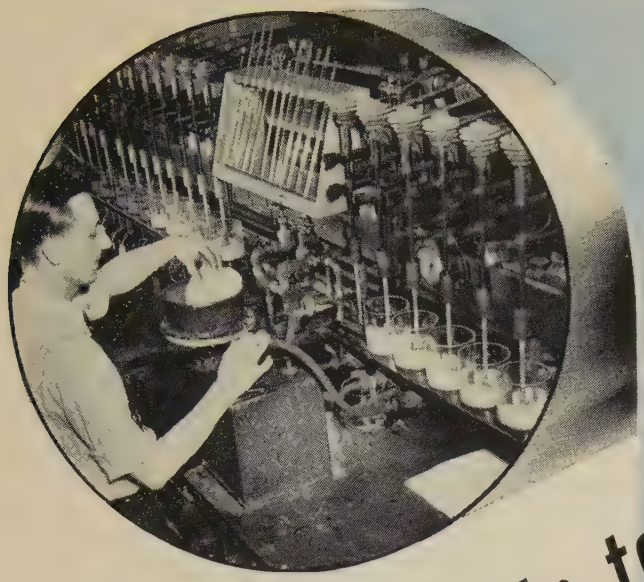

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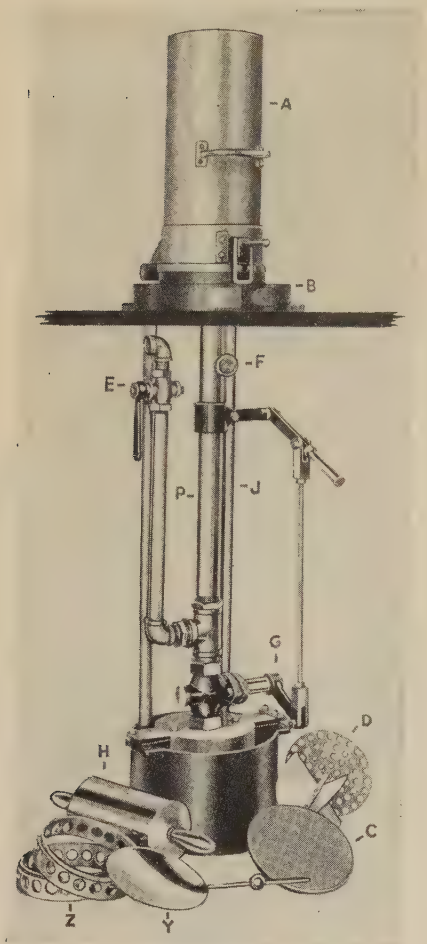
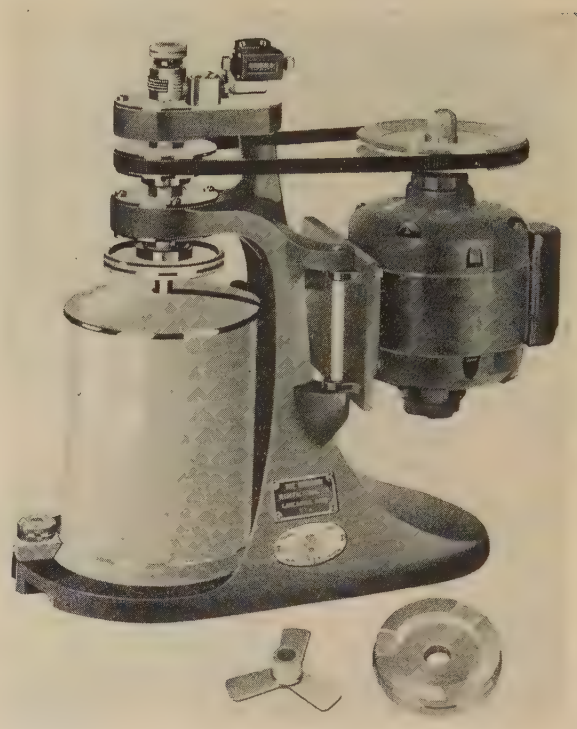
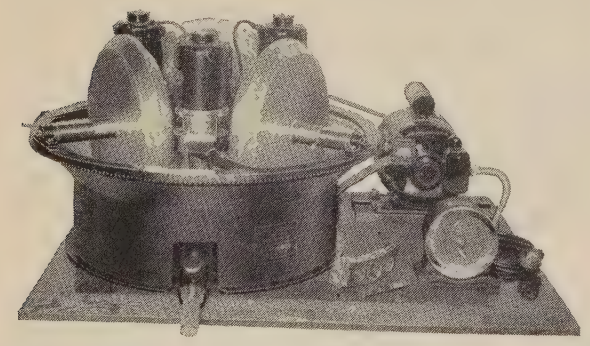


*SHEET MACHINE—DRYER RINGS—DISCS



*DISINTEGRATOR



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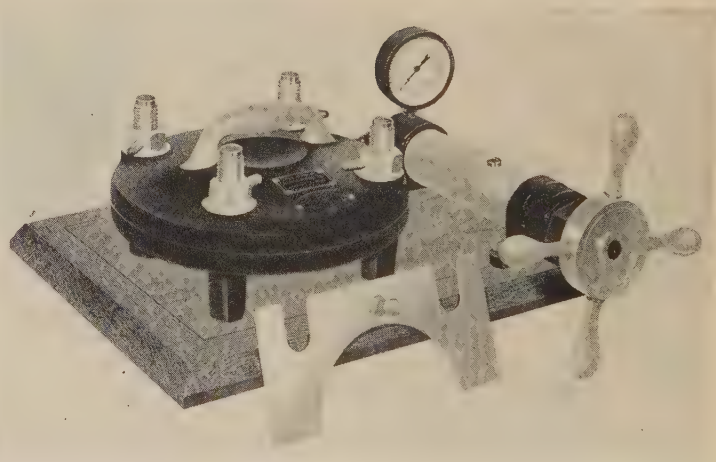

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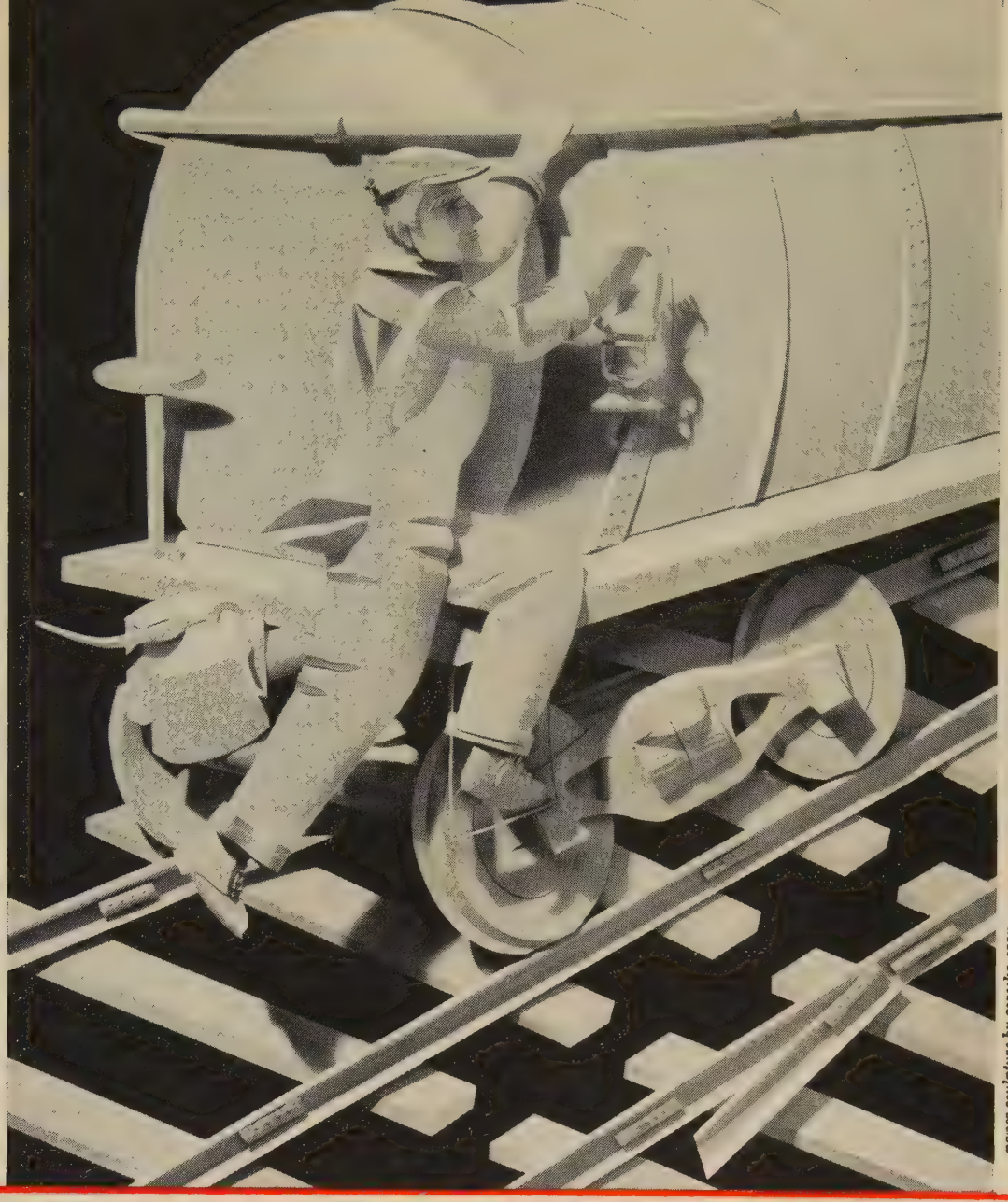

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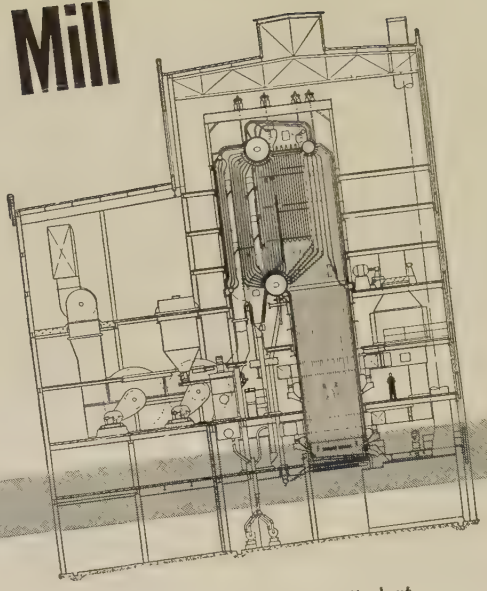

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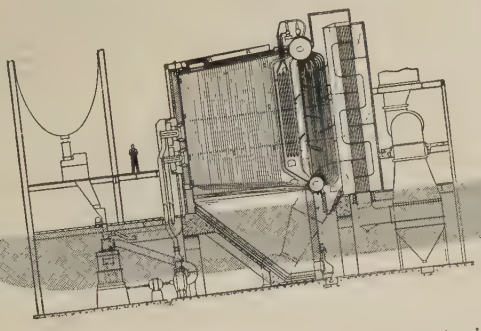

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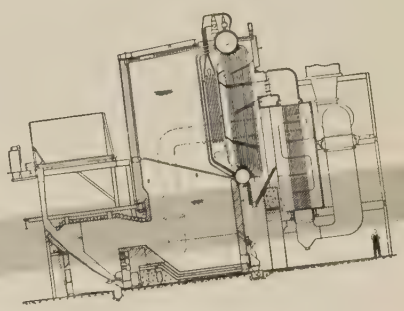

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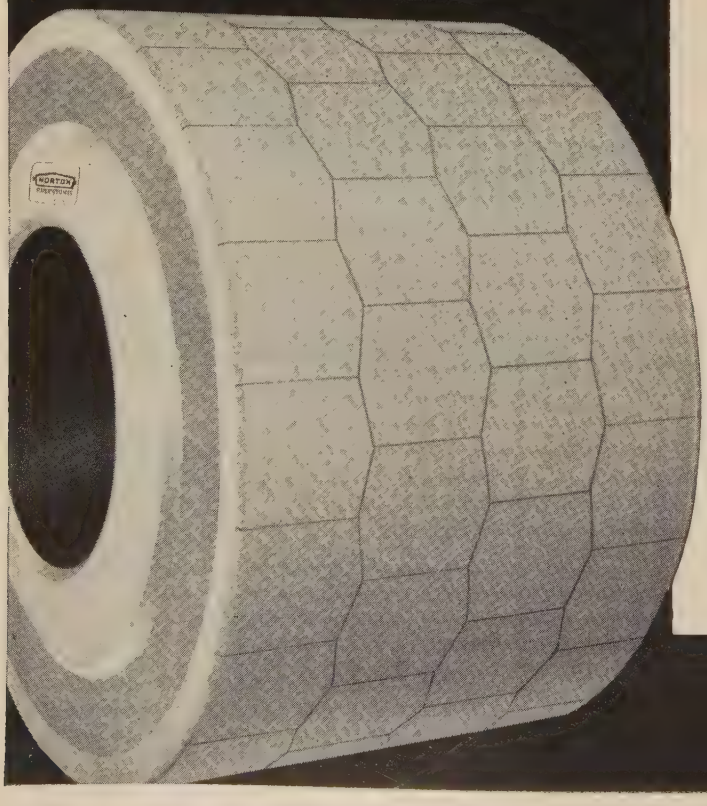

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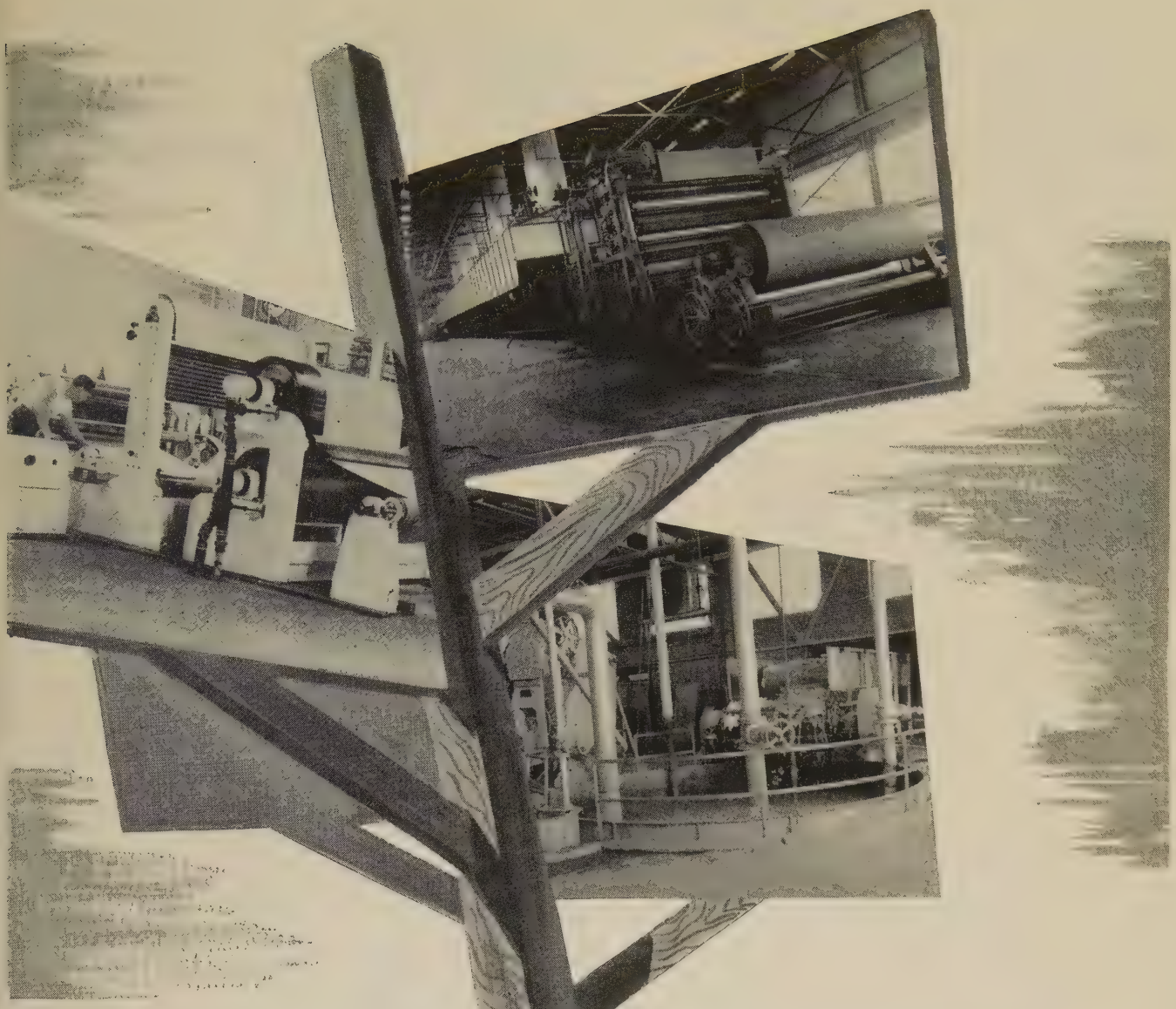

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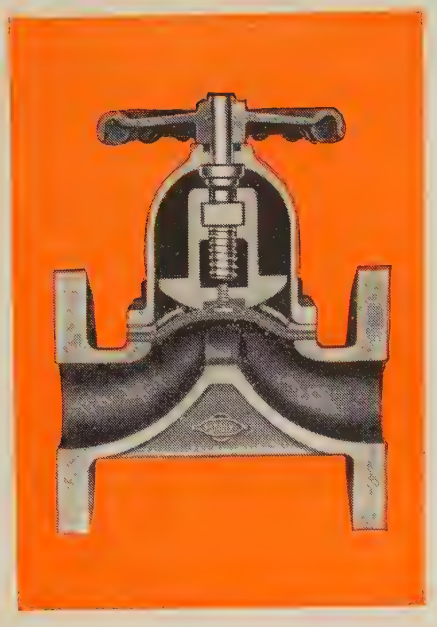

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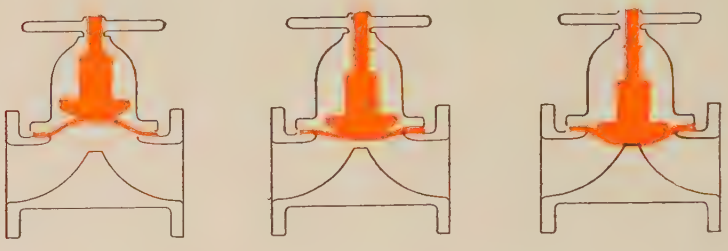

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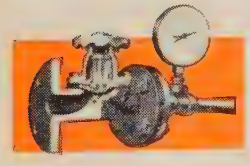

Diaphragm gives leak-tight closure against grit, scale, solid matter . . . Even when something as substantial as an 8-penny nail is trapped on the weir of a rubber lined valve, as illustrated, the resilient diaphragm, plus the large area of contact, gives leak-tight closure against pressure or vacuum.

Diaphragm lifts high for streamlined flow in either direction . . . Smooth, streamlined passage, without pockets, prevents trapping of sludge and reduces frictional resistance to a minimum—irrespective of direction of fluid flow. No disc holder in fluid stream. Grinnell-Saunders Diaphragm Valves are self-draining when installed with the spindle at 15 degrees above the horizontal position.

Diaphragm absolutely isolates working parts from fluid . . . There's no "if" about the way a continuous, one-piece diaphragm seals off the working parts from fluids; no perforation or puncture in the diaphragm where fluid or gas can possibly leak by the valve spindle. No sticking, clogging or corroding of working parts. Valve lubricant cannot contaminate the fluid in the line.

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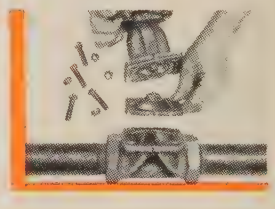

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Selection of Steam Pressures and Temperatures for Paper Mill Power Plants

M. H. DIXON, W. C. BLOOMQUIST, and W. B. WILSON

The purpose of this paper is to present various performance data which will enable the paper mill power engineer to select the proper turbine or combination of turbines to give optimum economy and operating flexibility. The data should be particularly useful in studying the mill heat balance to secure the proper relation between electric power and process steam. From these data, steam flows, exhaust and extraction temperatures, and electric energy produced by steam turbine-generators for paper mill power plants can be readily determined. By use of these data the performance of condensing, noncondensing, and extraction-type turbines can be estimated for a range of initial and process steam pressures.

THE authors fully appreciate that it is impossible to cover a subject as broad as this one in a single paper. Such a paper would have to include initial investment and operating costs, load factor, daily load curve, heat balance, and various operating problems. The authors have limited their presentation to data for the selection of the proper turbine or combination of turbines for optimum operating economy.

The proper selection of initial steam pressure and temperature and the application of turbines to provide maximum economies can be made only after a complete analysis of the steam and electric requirements—heat balance—has been made for the particular mill.

One problem that presents the greatest complication to power engineers is the relative over-all efficiency of the numerous combinations of boiler and turbine pressures and temperatures and the type of turbine to select. The usual methods of performance calculations are too laborious for necessary preliminary studies.

The main purpose of this paper is to present estimating data that can be used to arrive at the proper steam conditions and type of turbine quickly. These estimating data, sufficiently accurate for preliminary studies, will serve as a tool for the power engineer to check the effect of different steam conditions for his own plant. The data should be especially useful for new mills and for existing mills contemplating the addition or replacement of steam and power equipment. Also, some of the information may serve as a clue to methods of improving present operations.

The power plant must have sufficient flexibility so a maximum amount of electric power can be generated from the process steam requirements. Except in isolated cases where the paper mill has provision for interchange of power with the utility, the use of steam and electric energy cannot always be maintained in perfect balance. Winter heating loads, batch processes, and normal daily variations in the process requirements will all act to upset a perfect balance.

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To cover the range of variable demand for process steam and electric energy, condensing-extraction turbines, or a combination of condensing and noncondensing turbines offer a number of advantages in satisfying these variable demands without attention from the operator. By proper application and adjustment of the turbine governing mechanisms, such a combination of turbines will generate the required electric power and supply the process steam requirements with a minimum amount of steam being passed to the condensers.

OVER-ALL POWER PLANT THERMAL EFFICIENCY

The cost of steam and power for paper mills ranges from 10 to 20% of the cost of the product—a good average is around 15%. Even though this 15% is a small part of the total, the mill fixed expenses are high so even a few per cent saving in fuel for the steam and power exerts a leverage action on the net earnings of the company and is therefore of interest to the power engineer.

A power engineer is interested in the minimum cost of both steam and electric power. The concept of over-all plant thermal efficiency—a measure of the fuel cost—is the most straight-forward method of determining the cost of steam and power generation and is a good basis for comparing costs for various methods of producing steam and power. Over-all plant thermal efficiency is defined as follows:*

$$\text{Efficiency} = \frac{\text{Total available and useful output}}{\text{Total actual input}}$$

which then clearly recognizes both steam, or other forms of heat energy, and electric power; the useful electric power output can be converted into an equivalent heat basis.

The power-plant thermal efficiency of paper mills

* This expression cannot be used directly as a measure of over-all fuel costs when a part or all of the electric power is purchased.

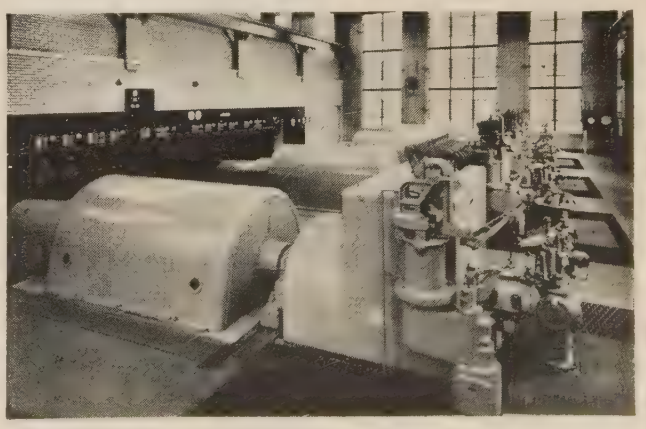

Fig. 1. Four 5000-kw. noncondensing turbine generators of a type widely used in paper mills

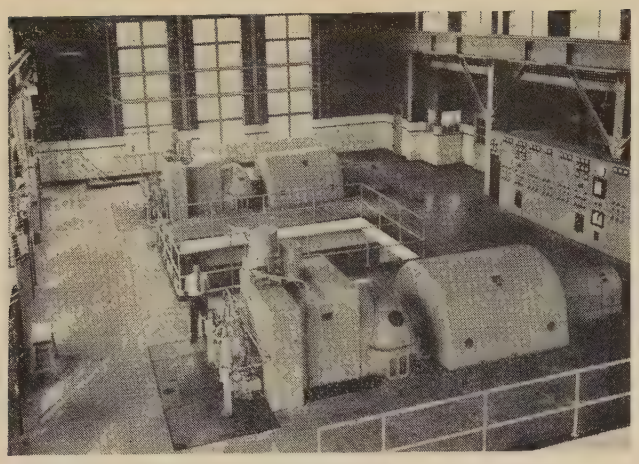

Fig. 2. Two 5000-kw., double automatic extraction-condensing turbine generators which supply process steam at two different pressures

ranges from 35 to 75%, depending upon the relationship of by-product power and condensing power; a practical maximum power-plant thermal efficiency is 70 to 80%.

The noncondensing turbine plant has the lowest over-all thermal efficiency when there is no requirement for process steam but has the highest over-all thermal efficiency when utilized to generate power and exhaust all steam for process requirements. This type of turbine, shown in Fig. 1, has wide usage in the paper industry.

The condensing-extraction turbine, operated with high extraction flows, closely approaches the thermal efficiency of a noncondensing turbine and provides flexibility in supplying variable demands for process steam and electric power. Because of this operating flexibility, the condensing-extraction turbine as shown in Fig. 2 is widely used as a prime mover in the paper industry.

Figure 3 shows how the various types of turbines influence the efficiency. The effect on thermal efficiency when operating condensing-extraction or a combination of condensing, noncondensing, and condensing-

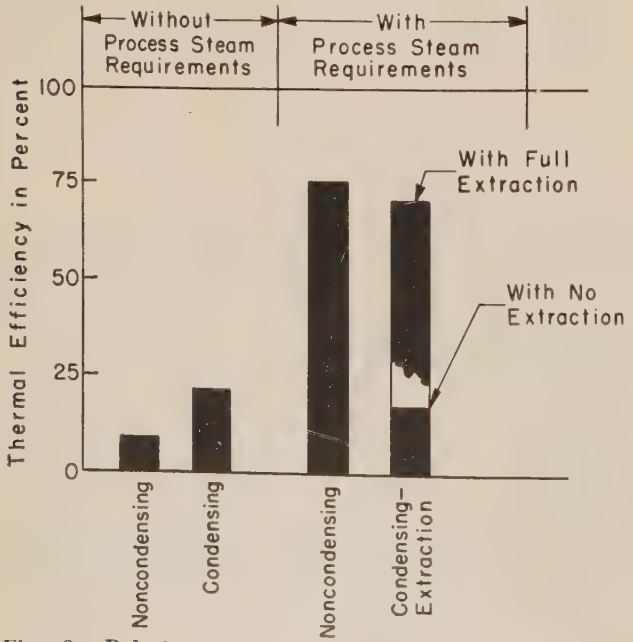

Fig. 3. Relative power-plant thermal efficiencies for different methods of steam-turbine operation

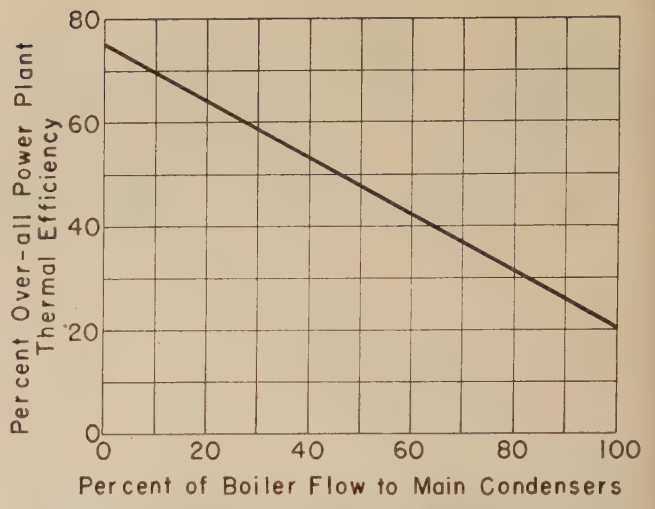

Fig. 4. Approximate over-all thermal efficiency of power plants supplying electric power and process steam

extraction turbines with various amounts of steam flowing to the condenser is also shown in Fig. 4.

From Fig. 4 the engineer can quickly approximate the power-plant thermal efficiency and also predict the efficiency for changes in methods of operation, such as the addition of a condensing or noncondensing turbine, steam supplied direct to process, etc., without the laborious detail calculations usually required. Figure 4 is based on the following: (1) at zero flow to the condenser, boiler efficiency of 85%, auxiliary and miscellaneous power of 7%, and unrecovered turbine and generator losses of 5%, which corresponds to a plant thermal efficiency of 75%; (2) at 100% flow to the condenser, a plant heat rate of 17,000 B.t.u. per kw.-hr., which corresponds to a plant thermal efficiency of 20%.

Example No. 1 (Illustrating Use of Fig. 4)

A mill has a total boiler output of 175,000 lb. per hr.; 135,000 lb. per hr. is used for by-product power genera-

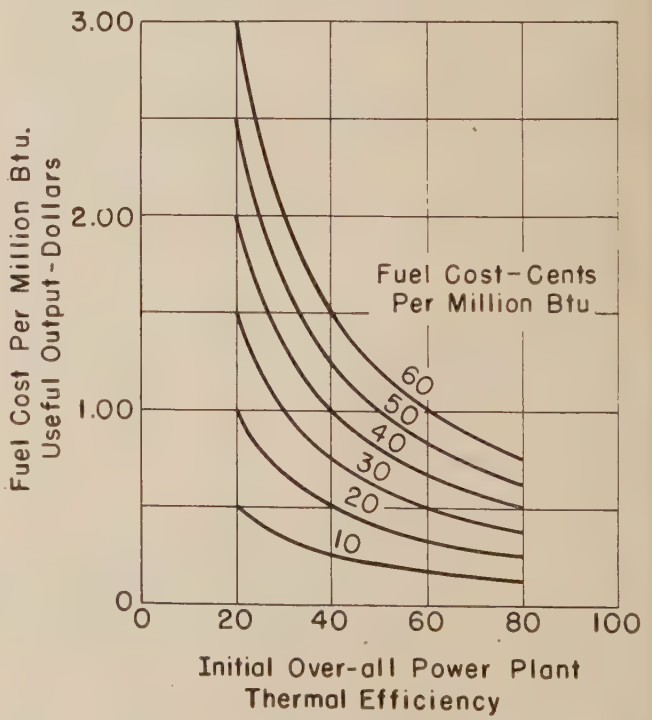

Fig. 5. Fuel cost per million B.t.u. useful output in terms of power-plant thermal efficiency

tion and process work, steam driven auxiliaries exhausting to process, etc.; 40,000 lb. per hr. goes to the main condensers for power generation. What is the approximate thermal efficiency? The per cent of boiler flow to the main condensers is:

$$\frac{40{,}000}{175{,}000} = 0.23 \text{ or } 23\%$$

From this curve the plant thermal efficiency is 62%.

Example No. 2 (Illustrating Use of Fig. 5)

A mill is operating at a relatively low power-plant thermal efficiency of 40%. By producing more by-product power and obtaining a better heat balance, the thermal efficiency is improved to 60%.

What is the yearly saving if fuel costs 40 cents per million B.t.u.?

From Fig. 5 the saving in fuel cost per million B.t.u. useful output is: \$1.00 − \$0.67 = \$0.33.

If the average output is 100,000 lb. per hr. for 7500 hours operation per year and assuming 1 million useful B.t.u. per 1000 pounds of steam, the annual saving is:

$$\$0.33 \times 7500 \times \frac{100{,}000}{1000} = \$250{,}000$$

CASE STUDIES ILLUSTRATING USE OF DATA

In paper mills, the process steam pressure is generally established to deliver heat at the required temperature. The quantity (pounds per hour) is determined by the heat, B.t.u., that must be delivered to the process; the temperature of the steam leaving the turbines is usually selected to supply saturated or slightly superheated steam at the process unless provision is made for desuperheating. Its determination will further be effected by the use of electric drives or back-pressure turbines for motive power on paper machines and by the limitations of the steam-distribution system within the mill.

It would be desirable to have complete turbine performance data for the various types of turbines and for the various steam conditions for each method of supplying the mill process steam and electric power requirements. However, from a practical standpoint that is too laborious when many alternatives are considered. Although the data presented here are for full-load machine rating only, they are applicable for the various types of turbines. From these data the most attractive operating cycles can be quickly determined; the final selection can be arrived at by using more complete performance data calculated for the few alternates remaining.

To illustrate the use of the data several examples are worked out in detail. A summary of various

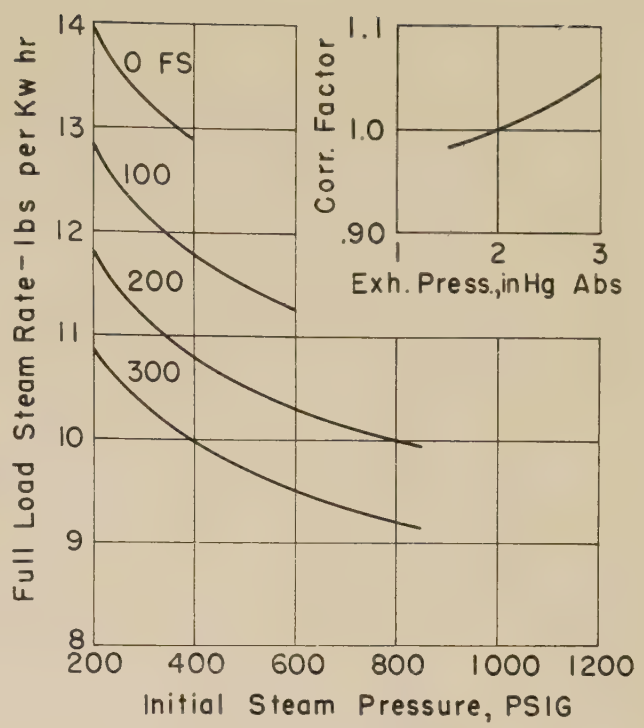

Fig. 6. Estimated steam rates for 5000-kw., 3600-r.p.m., condensing, turbine-generator units

methods of providing process steam and electric power is tabulated.

The comparison of the over-all plant thermal efficiency and relative fuel consumption for the various methods indicates the importance of such studies to establish the optimum cycle. Although these data pertain only to the over-all fuel usage, due allowance should be made for the initial and operating costs and existing mill pressures; the most efficient cycle may not necessarily be the most economical one.

The following examples (3–5) are based on a mill requiring 125,000 lb. per hr. process steam at 50 p.s.i.g. and 7500 kw. of electric power.

Example 3

Assume that the mill requirements are supplied by a 7500-kw. condensing-extraction turbine designed for steam conditions of 600 p.s.i.g.—750°F. (261°FS.)—2 in. Hg abs. with automatic extraction at 50 p.s.i.g.

A condensing-extraction turbine is in effect a combined noncondensing turbine and condensing turbine which has the extraction steam flowing in parallel with additional steam that passes through the turbine and exhausts to the condenser.

Comparison of Efficiency and Fuel Consumption of Different Plants

Power Plant Requirements: 7500 kw. Electric Energy; 125,000 lb. per hr. of Process Steam at 50 p.s.i.g.; Superheat of Extracted Steam Not Critical

Turbine-generators required and rating in kilowatts	*Turbine design steam conditions*	*Power generated in kilowatts by Process steam*	*Condensing steam*	*Per cent over-all power plant thermal efficiency*	*Relative fuel consumption*
1–7500	200 lb.—600°F.—2 in. Hg AE at 50 lb.	3070	4430	58	100
1–7500	400 lb.—650°F.—2 in. Hg AE at 50 lb.	4700	2800	64	90.6
1–7500	600 lb.—750°F.—2 in. Hg AE at 50 lb.	5760	1740	68	85.3
1–6000	600 lb.—750°F.—50 lb.	6000	1500	69	84.1
1–1500	600 lb.—750°F.—2 in. Hg				
1–7500	1200 lb.—825°F.—50 lb.	7500	0	75	77.4

The approximate performance of an extraction-type machine can be determined for estimating purposes from data included here by:

1. Determining the output of the required extraction steam flow as though it were passed through a noncondensing turbine having an exhaust pressure equal to the extraction pressure and then correcting the output (kilowatts) so obtained by the multiplying factor 0.95 from Fig. 8.

2. Finding deficit of kilowatts that must be produced by condensing power by subtracting the corrected output found in (1) above from the total required electric output.

3. Finding the steam rate, *SR*, for a straight condensing turbine corresponding to the initial pressure and temperature and the exhaust vacuum by using Figs. 6 and 9. Correct for turbine rating and for a condensing-extraction unit by using Fig. 8. Flow to the condenser will then be the corrected steam rate times the kilowatts produced by condensing power.

Now to continue with this example and to find the required boiler output, steam flow to the condenser, and the over-all power-plant thermal efficiency.

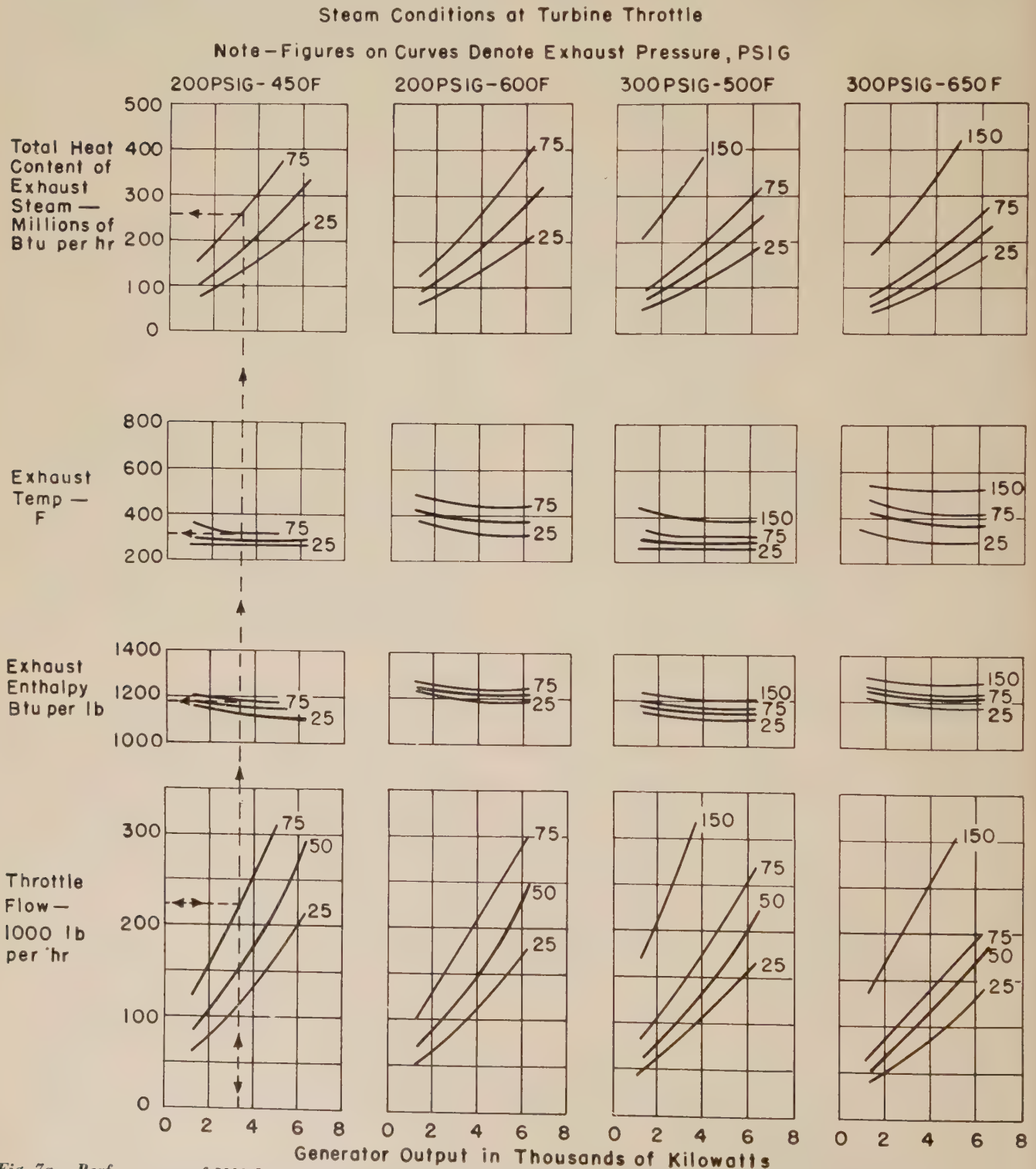
Steam Conditions at Turbine Throttle
Note—Figures on Curves Denote Exhaust Pressure, PSIG
200PSIG-450F
200PSIG-600F
300PSIG-500F
300PSIG-650F
Total Heat Content of Exhaust Steam — Millions of Btu per hr
Exhaust Temp — F
Exhaust Enthalpy Btu per lb
Throttle Flow — 1000 lb per hr
Generator Output in Thousands of Kilowatts

Fig. 7a. Performance of 5000-kw., 3600-r.p.m., noncondensing turbine-generator units for various initial and exhaust steam conditions. (See examples illustrating use of data.)

Step 1

Power generated by extraction steam flow:

Noncondensing output for a 5000-kw. unit (Fig. 7b,) at 600 p.s.i.g.—750°F.—50 p.s.i.g. and 125,000 lb. per hr. is 6000 kw.

Corrected noncondensing output for a 7500-kw. unit:

6000 kw. × 1.01 (factor at 7500 kw. from Fig. 8) = 6060 kw.

Corrected output from extraction flow for a 7500-kw. extraction unit:

6060 × 0.95 (factor from Fig. 8) = 5760 kw.

Step 2

Power to be generated by condensing steam flow:

7500 kw. − 5760 kw. = 1740 kw.

Step 3

Over-all (inlet-to-exhaust) steam rate:

SR of a 5000-kw. condensing-extraction unit, Figs. 6 and 8 at 600 p.s.i.g.—750°F.—2 in. Hg abs.:

9.8 × 1.06 = 10.4 lb. per kw.-hr.

SR of a 7500-kw. condensing-extraction unit:

10.4 × 0.99 (factor from Fig. 8) = 10.3 lb. per kw.-hr.

Steam flow to the condenser:

1740 kw. (step 2 above) × 10.3 = 17,900 lb. per hr.

Total boiler or throttle flow:

125,000 + 17,900 = 142,900 lb. per hr.

Per cent of boiler output passed to main condenser:

$\frac{17,900}{142,900}$ = 0.125 or 12.5%.

Over-all power-plant thermal efficiency: 68% as read from Fig. 4.

Turbine-generator required: One 7500-kw. condensing-

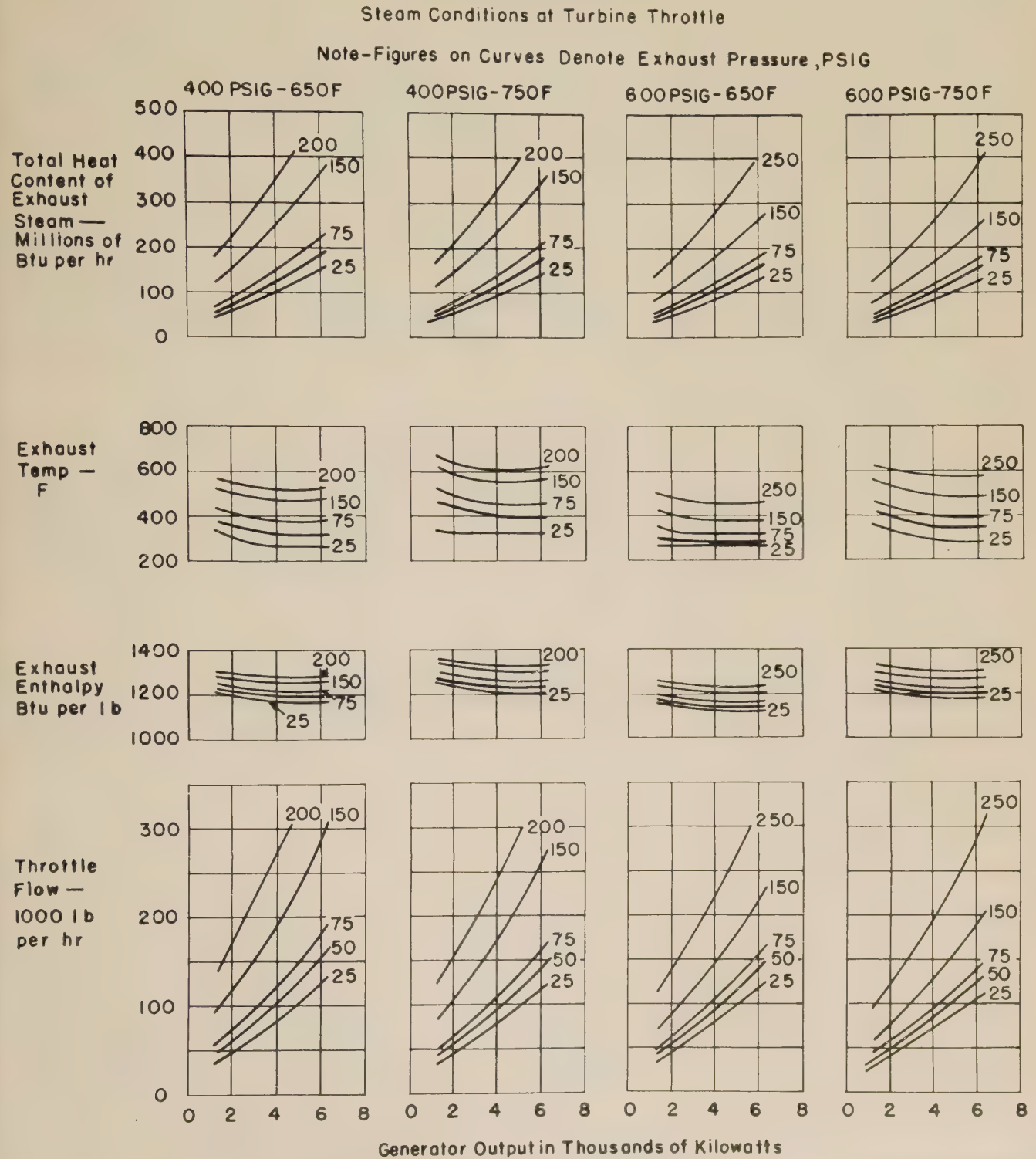
Steam Conditions at Turbine Throttle
Note-Figures on Curves Denote Exhaust Pressure, PSIG
400 PSIG-650F
400PSIG-750F
600PSIG-650F
600 PSIG-750F
Total Heat Content of Exhaust Steam — Millions of Btu per hr
Exhaust Temp — F
Exhaust Enthalpy Btu per lb
Throttle Flow — 1000 lb per hr
Generator Output in Thousands of Kilowatts

Fig. 7b. Performance of 5000-kw., 3600-r.p.m., noncondensing turbine-generator units for various initial and exhaust steam conditions. (See examples illustrating use of data.)

extraction unit designed for 600 p.s.i.g.—750°F.—2 in. Hg abs. with automatic extraction at 50 p.s.i.g.

Example 4

Assume mill requirements same as for example 3, and consider the use of a combination of a condensing turbine and a noncondensing turbine with initial steam conditions of 600 p.s.i.g., 750°F. (261°FS). The noncondensing turbine exhausts at 50 p.s.i.g. and the condensing turbine exhausts at 2 in. Hg abs.

Step 1

Power generated by process steam:

Referring to Fig. 7b, and neglecting the slight change in the multiplying factor (Fig. 8) between 5000-kw. and 6000-kw. ratings, a flow of 125,000 lb. per hr. at 600 p.s.i.g.—750°F.—50 p.s.i.g. will generate 6000 kw., which is equivalent to a steam rate of 20.8 lb. per kw.-hr.

Step 2

Power to be generated by condensing turbine:

7500 kw. − 6000 kw. = 1500 kw.

Steam rate for condensing turbine:

Referring to Fig. 6, the steam rate for a 5000-kw. condensing turbine (steam conditions 600 p.s.i.g.—750°F. (261° FS)-2 in. Hg abs. is 9.8 lb. per kw.-hr.

Referring to Fig. 8, the multiplying factor at 1500 kw. is 1.12.

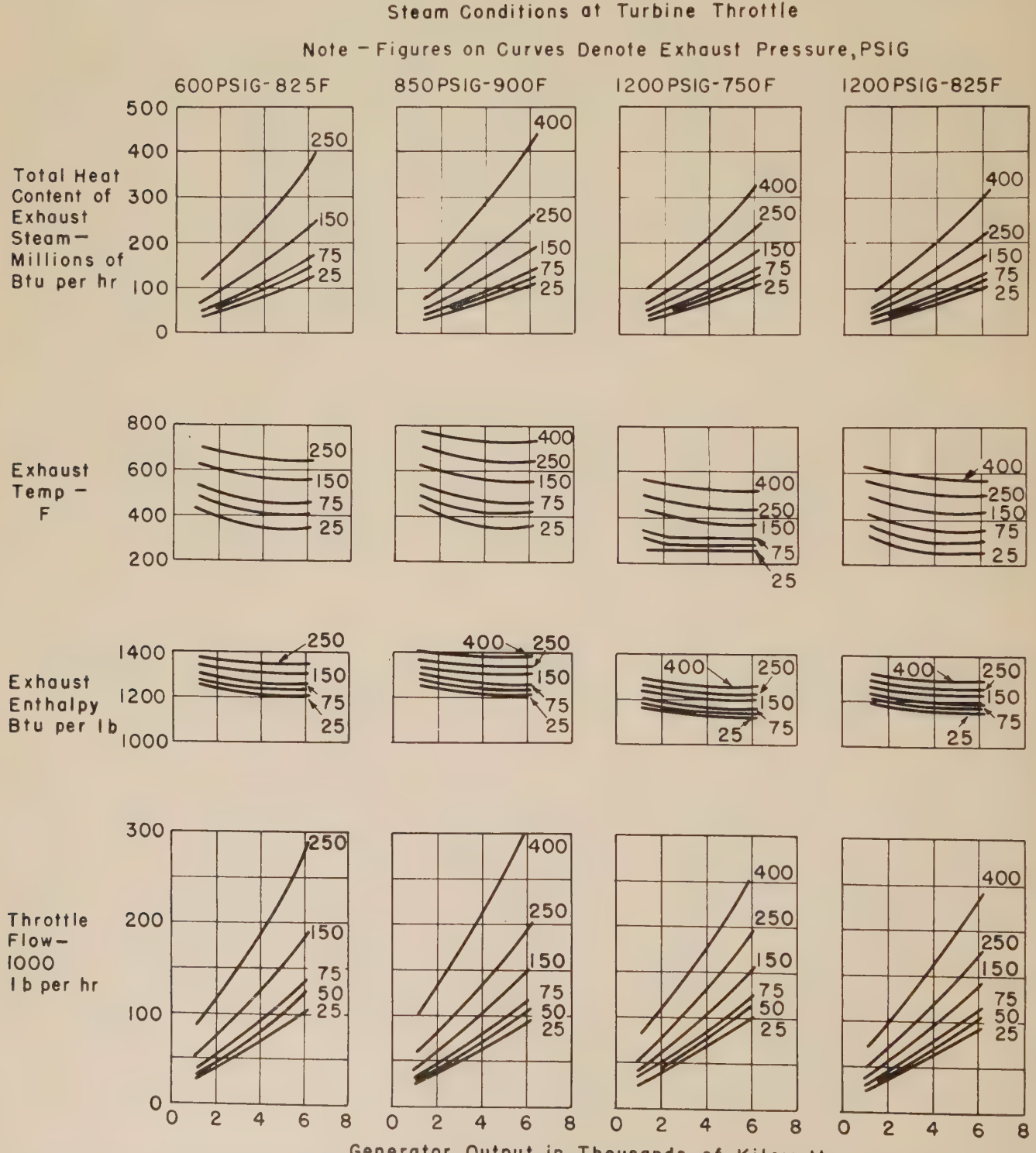
Steam Conditions at Turbine Throttle
Note – Figures on Curves Denote Exhaust Pressure, PSIG
600PSIG-825F
850PSIG-900F
1200PSIG-750F
1200PSIG-825F
Total Heat Content of Exhaust Steam – Millions of Btu per hr
Exhaust Temp – F
Exhaust Enthalpy Btu per lb
Throttle Flow – 1000 lb per hr
Generator Output in Thousands of Kilowatts

Fig. 7c. Performance of 5000-kw., 3600-r.p.m., noncondensing turbine-generator units for various initial and exhaust steam conditions. (See examples illustrating use of data.)

Steam rate for a 1500-kw. condensing turbine:
9.8 × 1.12 = 11.0 lb. per kw.-hr.
Steam flow for condensing turbine:
1500 kw. × 11.0 = 16,500 lb. per hr.
Total boiler or combined throttle flow:
125,000 + 16,500 = 141,500 lb. per hr.
Per cent of boiler output passed to main condenser:

$$\frac{16,500}{141,500} = 0.116 \text{ or } 11.6\%.$$

Over-all power-plant thermal efficiency:
69% as read from Fig. 4.
Turbine units required:
One 6000-kw. noncondensing unit designed for: 600 p.s.i.g. —750°F.—50 p.s.i.g.
One 1500-kw. condensing unit designed for: 600 p.s.i.g. —750°F.—2-in. Hg abs.

Example 5

Assume mill requirements same as for example 3, and consider increasing the initial steam conditions as required to generate all electric power by a noncondensing turbine having a steam flow equal to the process steam requirement.

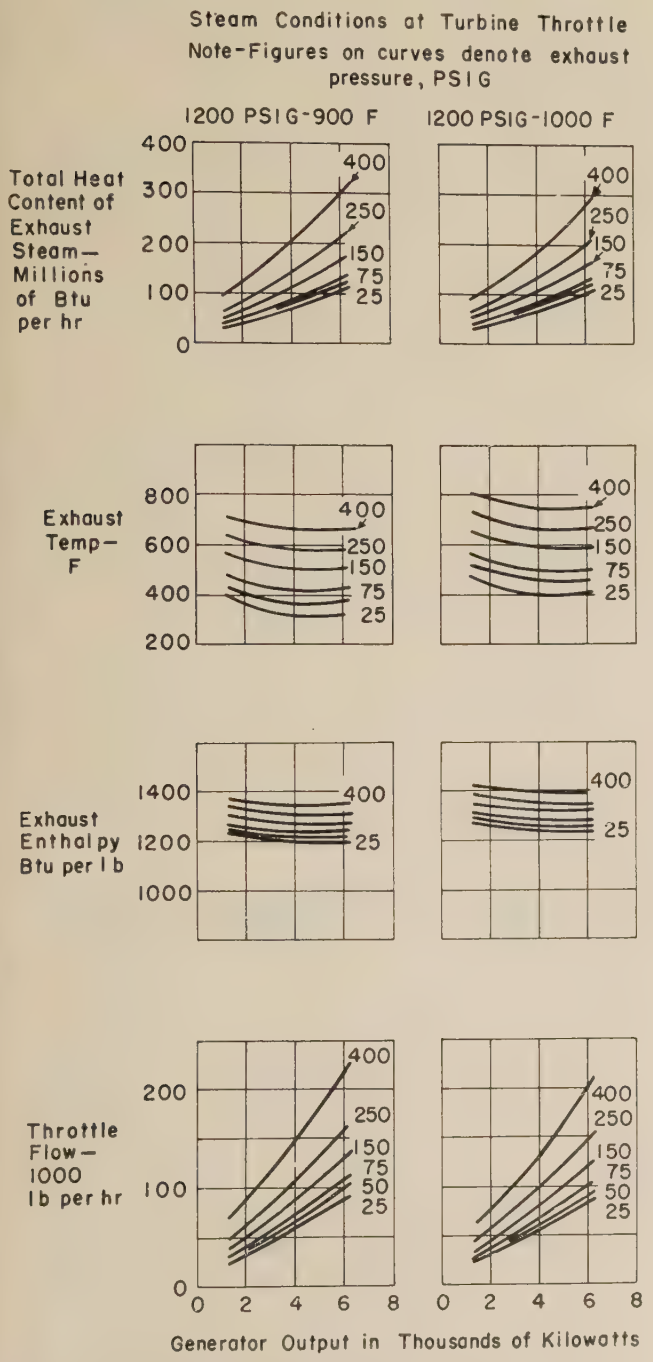
Steam Conditions at Turbine Throttle
Note-Figures on curves denote exhaust pressure, PSIG
1200 PSIG-900 F
1200 PSIG-1000 F
Total Heat Content of Exhaust Steam—Millions of Btu per hr
Exhaust Temp—F
Exhaust Enthalpy Btu per lb
Throttle Flow—1000 lb per hr
Generator Output in Thousands of Kilowatts

Fig. 7d. Performance of 5000-kw., 3600-r.p.m., noncondensing turbine-generator units for various initial and exhaust steam conditions. (See examples illustrating use of data.)

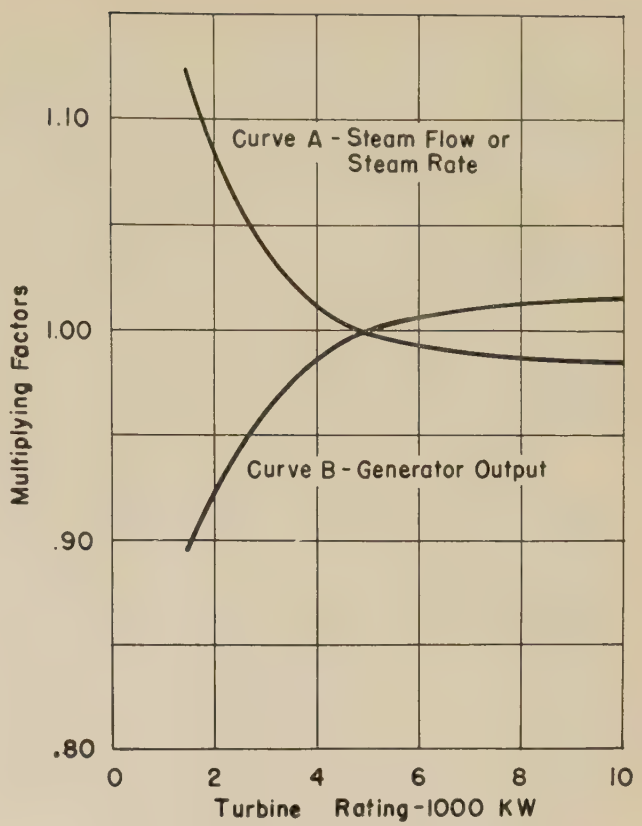

Fig. 8. Multiplying factors for various turbine ratings for use with Figs. 6 and 7. For automatic extraction turbines apply an additional factor of 1.06 for the steam rate and 0.95 for the generator output

To read the required initial steam conditions for the 7500-kw. unit, an equivalent flow for a 5000-kw. unit must be determined since the basic curves of Fig. 7 are for a 5000-kw. unit. This equivalent flow is equal to the required flow at 7500 kw. times the ratio of ratings times the ratio of multiplying factors for rating from Fig. 8.

Equivalent flow for a 5000-kw. unit:

$$125,000 \times \frac{5000}{7500} \times \frac{1.00}{0.99} = 84,000 \text{ lb. per hr.}$$

Required initial steam conditions:
Interpolation of Fig. 7c (read at 84,000 lb. per hr., 50 p.s.i.g. exhaust and 5000 kw.), indicates steam conditions of 1200 p.s.i.g.—825°F.—50 p.s.i.g. to generate required electric energy from the fixed process steam requirements.
Over-all power-plant thermal efficiency:
This is 75% as read from Fig. 4.
Turbine unit required:
One 7500-kw. noncondensing unit designed for: 1200 p.s.i.g.—825°F.—50 p.s.i.g.

Example 6

Assume mill requirements are 7500 kw. of electric energy and process steam at 25 p.s.i.g. The existing boiler plant delivers steam at 600 p.s.i.g.—825°F. What throttle flow is required to supply a 7500-kw. noncondensing turbine designed for 600 p.s.i.g.—825°F. —25 p.s.i.g.?

Full-load steam flow for a 5000-kw. unit:
From Fig. 7c read 84,000 lb. per hr.
Multiplying factor for steam rate at 7500 kw.:
From Fig. 8 read 0.99.

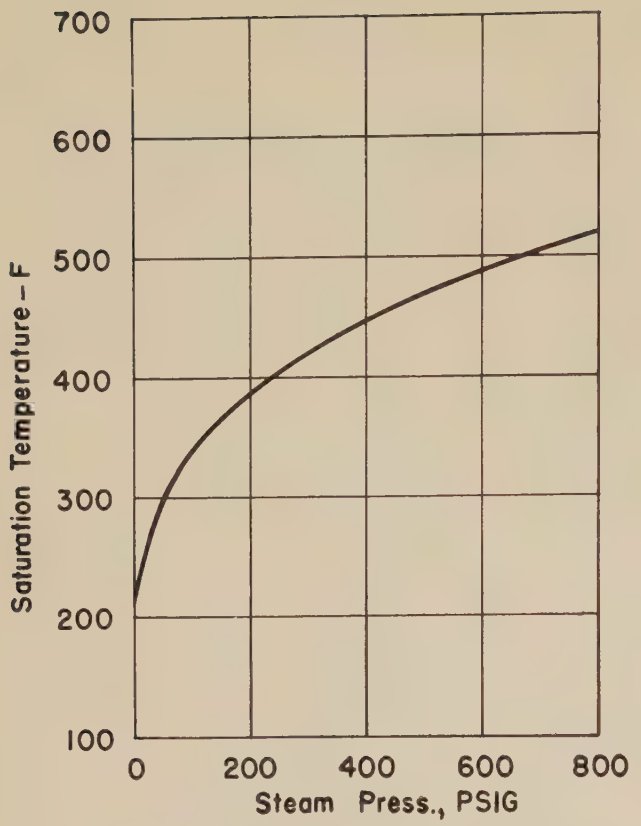

Fig. 9. Saturation temperature for steam at various pressures

Full-load steam flow for a 7500-kw. unit:

$$\text{Flow for a 5000-kw. unit} \times \frac{\text{desired rating}}{5000} \times \text{factor} =$$

$$84{,}000 \times \frac{7500}{5000} \times 0.99 = 125{,}000 \text{ lb. per hr.}$$

Approximate exhaust temperature and enthalpy (read at full-load conditions for a 5000-kw. unit):

From Fig. 7c read 340°F. and 1200 B.t.u. per lb.

Total heat content of process steam from a 7500-kw. unit: 125,000 × 1200 = 150,000,000 B.t.u. per hr.

Assuming that the plant requirements are 7500 kw. and 125,000 pounds of 25-p.s.i.g. process steam per hour, the initial conditions selected above give a perfect balance between steam and electric requirements. With such an ideal set of conditions, the over-all power-plant thermal efficiency will be 75% as read from Fig. 4. The electric energy is produced at this same efficiency which is equivalent to 4550 B.t.u. per kw.-hr.

Abbreviations

AE	=	Automatic extraction.
°F.	=	Degrees total temperature, Fahrenheit.
°FS	=	Degrees superheat, Fahrenheit.
in. Hg abs.	=	Inches of mercury, absolute.
p.s.i.g.	=	Pressure in pounds per square inch gage.
SR	=	Steam rate, pounds of steam per kilowatt-hour.

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A Stain for Use in the Microscopy of Beaten Fibers

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A two-color differential stain for papermaking fibers is described which employs a direct blue dye and a direct orange dye. Unbeaten fibers are stained blue while beaten fibers are stained varying mixtures of orange and blue depending on the degree of beating. The method is relatively independent of the kind of fiber. Staining is done directly on the microscope slide by a simple procedure.

The study of the microscopic appearance of fibers during the beating cycle affords much useful information in many cases. Fibrillation, length, proportion of debris, etc., all have an important bearing on the properties of the finished sheet, while indirect tests, such as freeness, seldom give a clear understanding as to what is happening in the stock preparation process.

Such studies can be made much more conveniently by the use of the differential stain here described. This stain and staining method are new as far as the author is aware although no claim is made that the literature has been rigorously combed for anticipatory ideas. It should also be pointed out that much still remains to be done to determine the full usefulness and the limitations of this stain. Cotton, cotton linters, linen, flax, ramie, hemp, and a few commonly used varieties of sulphite, sulphate, and soda pulps have been studied so far, including the cooked and uncooked or bleached and unbleached conditions for most of them. While the stain

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appears to be independent of these wide variations, it obviously must be checked by a number of different investigators.

Aniline dyes vary tremendously, of course, in their affinity for cellulose, and this affinity can vary with the character of the cellulose. Even the substantive or direct dyes vary widely for reasons which are not understood and these differences give rise to various methods of fiber identification with which all are familiar. Stains for determination of cooking degree, for bleachability, and for maturity of cotton have also been based on differences in affinity of dyes for cellulose of different characteristics.

The stain discussed here is for use under the microscope and is of the differential, two-color type. Insofar as it has been investigated it apparently responds to differences in fibrillation and extent of mechanical disintegration. Two direct dyes are used, one being an orange of Color Index No. 621 and the other a blue of Index No. 518.

Unbeaten fibers under this stain are almost pure blue, but in beaten stocks the fibrils, fiber debris, and bruised spots on the fibers stain orange. In other words, the orange of this two-component stain preferentially colors any part of the fiber that has been broken down or is mechanically damaged. In very heavily beaten stocks and in rebeaten broke there are also occasional fibers which are stained entirely orange or a greenish orange.

Successive samples taken from a beater and stained by this method will give an excellent picture of what is happening to the fibers during the beating cycle. As soon as fibrils appear they stain completely orange while the parent fibers remain blue. Fiber ends that are "broomed" will stain orange, the color fading into the blue of the rest of the fiber. Similarly, a crushed or bruised spot on a fiber will stain orange with a zone of mixed transition colors on either side and the remainder of the fiber blue.

The technique of applying the stain is simple and the conditions required are not too critical. Pontamine Sky Blue 6BX Greenish (C. I. No. 518) and Pontamine Fast Orange 6RN Conc. (C. I. No. 621) may be taken as prototypes of the two dyes. For the staining of most wood pulps a solution containing equal parts of the two dyes is satisfactory; for rag stocks, better results are obtained if the orange is in slight excess. The total strength of the staining solution, adding both dyes together, should be about 1.0%, and the solvent is distilled water.

It is convenient to have on hand a separate 1% stock solution of each dye. Then for wood pulps equal quantities can be mixed and for rag stocks about 45% blue to 55% orange. Working in this way, the ratio can be varied as needed but the final concentration of the staining mixture remains at 1.0%. Most workers will wish to vary the ratio to suit their own ideas of desirable contrast.

Given the stain solution, the staining procedure may be summarized as follows.

1. Prepare the fiber slide in any manner similar to TAPPI Standard T 401 wherein an evenly dispersed field is produced and dried down on the slide. Either slush pulp or finished paper may be the starting point. Melamine-treated papers should be cooked with a little alum first.
2. Add 6 to 8 drops of the staining solution to the fiber area on the slide, or enough to flood the fibers thoroughly but without over-flowing the slide.
3. Place the slide, with the stain, on a 60 °C. hot plate, such as is used to prepare slides according to the TAPPI method. Somewhat higher temperatures (70 °C.) are to be preferred but the regular 60 °C. hot plate is suggested for those so equipped. An infrared lamp, vertically supported, can be easily adjusted for this use.
4. Allow the staining solution to evaporate to dryness, which should be a matter of 5 to 10 minutes.
5. Wash, either in running water or by dipping, or by any convenient method that does not dislodge the fibers. Tap water, if not too hard, is satisfactory.
6. Dry and examine. The colors are reasonably permanent without a mounting medium. Water, glycerine, and methylene iodide destroy the colors slowly while Canada balsam and similar materials have an optical effect on the colors. Far more detail can be observed if no mounting material is used.

There are certain comments which should be made. In the first place almost any of the dyes in the two named Index numbers may be used and we should mention, as examples, Brilliant Benzo Blue 6BA (General Dyestuffs), Diphenyl Brilliant Blue FF (Geigy), Pheno Sky Blue 6BX (Calco), Pontamine Sky Blue 6BX (Du Pont), Direct Fast Orange MR (General Dyestuffs), Polyphenyl Orange 2R (Geigy), and Pontamine Fast Orange MRL (Du Pont). There are small differences in shade and in brightness among these various makes. At the moment the author slightly prefers Pontamine Sky Blue 6BX Greenish (Du Pont) or Brilliant Benzo Blue 6BA (General Dyestuffs) for the blue and either Pontamine Fast Orange 6RN (Du Pont) or Polyphenyl Orange 2R (Geigy) for the orange.

It is rather remarkable that the orange should be so closely limited as to type and chemical constitution. A great many yellows, oranges, and reds have been tried but only those of Color Index Nos. 620, 621, and 622 give this differential staining. Numbers 620 and 622 are both yellows and while they work, the color contrast is not as good as with the deeper-hued No. 621 orange. These three Index numbers represent dyes that are very closely related chemically.

The No. 518 blue is familiar to many for its use in identifying rayons. However, it is not quite so specific as the orange for this differential stain, and blues from Index Nos. 520, 516, 515, and 512 will work to some extent at least. As might be suspected, the dyes nearest in Index number to No. 518 give the best results.

Curiously enough, the commercial dyes seem to be just as useful as any specially purified product. The Du Pont Co. and the General Dyestuffs Corp. very kindly supplied the author with colors free from the usual salt or sodium sulphate loading and also recrystallized. Comparisons so far made do not indicate any great advantage for the latter.

Similarly, materials such as salt or sodium sulphate when added to the stain do not seem to effect its action. Even phenol and triethylene glycol in small quantities have only slight effect in most cases. Acids and alkalies, however, tend to destroy the differential feature.

Heat seems to be necessary for the orange dye to function properly. Staining can be done in the cold but the time required is very long and the results usually inconsistant. While a hot bath can be used, the technique of evaporating to dryness is a much easier procedure. The actual temperature used does not seem to be

critical, but it should be at least 60°C. if the orange is to show up well. Higher temperatures up to 85°C. shorten the time required, but above this the orange may be somewhat accentuated.

If an evaporation technique such as this is successful it can be suspected that actual concentration of the stain is not too critical and such is the case. The total concentration (both dyes together) may vary from 0.5 to 1.5% without much change except in total depth and even this difference is small. The 1.0% specified in the procedure gives about the right depth for most purposes, however, and has been used by the author in control and research for some time. Both the individual dye solutions and the mixed stain have good keeping qualities.

This stain is unusual in many respects and it raises many questions which time and further work may answer. It is, for instance, rather striking that it behaves equally well on such widely differing types of fibers as bleached cotton and unbleached sulphite.

Whether the orange component of this stain indicates and shows actual hydration depends on one's definitions. However, the stain does show definitely and clearly the effects of mechanical disintegration or mechanical work done on the fibers and this, of course, is a prerequisite to hydration. It is also notable that in the case of cotton fibers hydrated by mechanically gentle methods this stain, under optimum conditions, will show by differential coloring the fibrils in their angular position right in the fiber. This would indicate that under certain beating conditions the entire fibril structure is loosened or opened up, even though the fiber as a whole has not been bent, broken, or directly damaged by mechanical impact.

To some extent the orange component of the stain also seems to dye preferentially the thin-walled fibers, or to put it another way, the thin-walled fibers stain orange with a minimum of beating. Similarly, certain areas in wood pulp fibers likewise tend to absorb the orange dye after very little or almost no beating. Such observations might suggest a "skin" effect but they are more likely an indication that dye absorption is related to thickness of wall and degree of crystallinity, or to other characteristics related to "hydration." This view is supported by the fact that cotton linters, which are thick-walled and difficult to hydrate, stain a somewhat deeper blue than long staple cotton, and seldom take up any of the orange dye except in the fully-separated fibrils. Furthermore, viscose rayon, which is practically impossible to "hydrate" by papermaking methods, stains a clear deep blue even after the most prolonged beating. The fact that rayon swells readily in water and is less crystalline and less oriented than native cellulose, leads to some interesting speculations.

All of these various observations suggest that further studies of this combination of orange and blue dyes can lead to a better understanding of fiber structure and of those fiber properties such as crystallinity and orientation which may be related to "hydration" and papermaking qualities in general.

It was expected that eventually a single source of the the two dyes used in this stain would be needed when its use developed to the point of making exact comparisons. Prepublication interest has been so great, however, that this arrangement has now been made. Specially prepared dyes may be ordered from the Du Pont Co. under the names: Pontamine Sky Blue 6BX Greenish Special and Pontamine Fast Orange 6RN Special.

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The Reverse Roll Principle of Coating

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The reverse roll method of coating is described and its advantages mentioned. In the present study the coverages obtained on a 10-inch wide laboratory reverse roll coater were measured for a glue solution (Newtonian), a vinyl resin in organic solvent (Newtonian), a clay-casein coating color (plastic), a vinyl resin in organic solvent (pseudoplastic), and an aqueous dispersion of vinyl resin (thixotropic). The results are presented for different casting roll-backing roll speed ratios, nip clearances, and operating speeds. In general, the wet thickness (t) applied to the web appears proportional to the metering roll clearance (D) and the casting roll-backing roll speed ratio (R) and is independent of web speed as long as this ratio remains constant. The coverage can be predicted with a fair degree of accuracy by the relation $t = kDR$ where k is a constant. For aqueous solutions with the particular metering roll-casting roll speed ratio used (0.33), k had a value of about 0.5. The value of k is affected significantly by the metering roll-casting roll speed ratio, increasing as this variable decreases. Below a certain critical value of R, in no case found to be greater than one, the coating becomes nonuniform.

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The reverse-roll coater is one of many types of coating machines (*1*). The advantages of the reverse-roll coater are that a wide range of coating viscosities can be applied and even distribution can be obtained on webs of irregular surface. The principle of the machine is covered by a patent (*2*) now expired. The nomenclature of reverse-roll coater was derived apparently from the reverse direction of the moving surfaces of the casting roll and backing roll.

The coater consists of two or, preferably, three rolls all driven in the same angular direction. The two roll unit consists of a backing roll which is a composition, resilient roll and a polished metal roll which is referred to as a casting roll. The casting roll picks up the coating from a pan and the coating is metered

Table I. Viscosimeter Measurements on Vinyl Resin Hydrosol XDH5005 (Temp. 83° F. No. 1 Brookfield Viscosimeter Spindle)

R.p.m.	Apparent viscosity, cp.							
	Cycle 1		Cycle 2		Cycle 3		Cycle 4	
	Decreasing	Increasing	Decreasing	Increasing	Decreasing	Increasing	Decreasing	Increasing
20	215	320	...	320	...	330	...	320
10	300	440	314	420	330	...	330	420
4	425	750	475	620	500	655	465	620
2	...	1060	...	810	...	840	...	780

Properties of the colors are summarized in Table II.

by an adjustable doctor blade set at a desired gap. The metered coating is then wiped onto the web. There is a slight pressure exerted on the web running between the backing roll and the casting roll. The fixed doctor blade for metering the coating has the disadvantage of causing streaks in the coating due to the fact that foreign particles lodge between the doctor and the casting roll. The addition of a third roll, referred to as the metering roll, provides a moving doctoring surface of adjustable clearance to meter the coating. This roll is provided with a scraping doctor to clean continuously its peripheral surface. The machine can be adapted to a fountain supply if desired.

Although it is generally known that the coating applied is directly proportional to the gap between the metering and casting roll and to the ratio of casting roll speed to web speed, no information has been published on a quantitative basis.

The purpose of this paper is to attempt to supply some information along these lines.

The experiments were run on a 10-inch wide laboratory reverse-roll coater. Paper was used as a web material because of availability and economy.

COATING COLORS

In order to span the different types of coating "colors" which might be used on the reverse-roll coater and to study also colors exhibiting different rheological behavior, five materials were selected. These were on an aqueous gelatin solution, a typical casein-clay coating color, a vinyl resin solution, a vinyl resin hydrosol, and a vinyl resin organosol. Their solid contents were, respectively, 14.8, 39.78, 35.0, 51.0, and 63.0%. Their rheological characteristics were measured on a Brookfield viscosimeter. The gelatin solution was essentially Newtonian having an apparent viscosity independent of rate of shear, the casein-clay color was plastic showing a yield value, the vinyl resin XL5426 was only slightly pseudoplastic, while the vinyl resin XDL5065 was exceedingly pseudoplastic as indicated by a pronounced bowing of the curve. The vinyl resin hydrosol was extremely thixotropic, as shown by Table I.

EXPERIMENTAL

Before commencing a run, the coating color was brought to temperature usually that of the room and coating machine. The metering roll-casting roll gap was set to the desired value with the aid of a feeler gage. The suspension was then poured into the reservoir formed by the metering and casting rolls, the paper threaded and a run commenced. When conditions became steady, the speeds of the casting and backing rolls were measured and at least two samples of the coating were taken at the winder. The samples, together with samples of the raw stock, were conditioned for 2 or more days at 65% R.H. and 70° F. Coverage was then determined by cutting 8-inch squares from raw stock and coated paper and obtaining the difference in weight.

Speeds are believed to be accurate to ±2%; coverages to ±7% for the thinnest coatings, ±5% for the thicker. Errors may be due to variation in basis weight of the coating stock, incomplete volatilization of solvents, etc., as well as errors in measurement.

It was noted that, if, at constant paper or backing-roll speed, the casting-roll speed was steadily reduced, a critical speed was reached at which a continuous, uniform coating was no longer obtained. This critical speed was determined for a few settings on each coating mixture.

A final series of runs were made with a different casein-clay coating to evaluate qualitatively the effects of metering roll: casting-roll speed ratio.

The results are given in Table III, which shows the surface speeds of the casting and backing rolls, the clearance, the ratio of the speeds, and the wet thickness of the coating. This last is calculated from the coverage knowing the specific gravity and per cent solids of the color. The metering roll speed was one third of the casting-roll speed throughout Table III.

Critical speeds for a number of coating mixes and conditions are shown in Table IV. The casting roll speed, in every case, is the lowest speed at which a continuous coating could be obtained. Any further drop in casting-roll speed led to streaks, dot formation, or other irregularities.

DISCUSSION

Study of Table III indicates that for any given solution the coverage varies directly with the metering roll clearance and the casting roll-backing roll speed ratio and is independent of web speed as long as this ratio remains constant.

Throughout the above experiments the metering roll-casting roll speed ratio was constant (0.33); hence for a given coating color the wet thickness t_1 on the casting roll might be expected to be given by:

$$t_1 = kD$$

Table II

Color	Per cent solids	Specific gravity	Apparent viscosity at 10 r.p.m., cp. (Brookfield)	
Gelatin	14.8	1.00	34	(90° F.)
Casein-clay	39.78	1.29	128	(75° F.)
Vinyl resin XDL5065	63.0	1.05	5280	(70° F.)
Vinyl resin XL5426	35.0	1.00	3300	(70° F.)
Vinyl resin XDH5005	51.3	1.00	330–420	(70° F.)

Two paper stocks were used; for the gelatin coatings a lightweight 43-pound stock, for the other colors a heavier coating stock of 69 pound basis weight (25 × 40—500).

Table III. Results

Color	Backing roll speed, f.p.m.	Casting roll speed, f.p.m.	Ratio, roll speeds (R)	Clearance (D) in.	Coverage, lb./1000 sq. ft.	Wet thickness (t)	t/DR
Gelatin, 90° F.	9.4	9.4	1.0	0.003	1.26	0.00164	0.55
	18.6	17.7	0.98	0.003	1.53	0.00199	0.68
	27.6	26.0	0.98	0.003	1.39	0.00181	0.63
	39.3	37.0	0.94	0.003	1.19	0.00155	0.55
	29.0	47.8	1.65	0.003	1.80	0.00233	0.47
	29.5	58.1	1.98	0.003	2.07	0.00270	0.46
	29.0	86.0	2.96	0.003	2.96	0.00384	0.43
	29.2	30.1	1.03	0.0015	0.57	0.00074	0.48
	29.5	30.5	1.03	0.002	0.74	0.00096	0.47
	29.5	30.5	1.03	0.004	1.59	0.00206	0.50
	29.5	30.0	1.02	0.003	1.05	0.00136	0.45
Casein-clay, 80° F.	9.4	9.4	1.00	0.003	4.04	0.00151	0.50
	19.15	21.1	1.10	0.003	4.80	0.00179	0.54
	29.5	29.9	1.01	0.003	3.78	0.00141	0.47
	38.2	39.6	1.04	0.003	3.89	0.00145	0.47
	29.5	43.4	1.47	0.003	5.46	0.00204	0.46
	29.5	59.8	2.02	0.003	7.25	0.00271	0.45
	28.6	84.5	2.95	0.003	10.58	0.00395	0.45
	28.6	30.3	1.06	0.0015	2.11	0.00079	0.51
	28.6	45.4	1.59	0.0015	3.18	0.00119	0.50
	28.6	60.9	2.13	0.0015	4.40	0.00164	0.51
	28.6	84.5	2.96	0.0015	5.50	0.00206	0.47
	30.1	30.9	1.03	0.002	3.08	0.00115	0.57
	29.5	60.1	2.04	0.002	5.19	0.00194	0.48
	29.5	84.5	2.86	0.002	7.73	0.00289	0.51
	28.8	30.5	1.06	0.004	5.37	0.00201	0.48
	28.8	47.2	1.64	0.004	7.53	0.00282	0.43
	28.2	61.5	2.18	0.004	10.38	0.00388	0.45
XL5426, 75° F.	8.95	8.55	0.96	0.003	4.62	0.00254	0.88
	18.5	18.3	0.99	0.003	4.01	0.00220	0.74
	29.2	30.7	1.05	0.003	3.70	0.00203	0.65
	39.4	43.5	1.10	0.003	4.81	0.00264	0.80
	14.45	15.5	1.07	0.003	3.22	0.00177	0.55
	14.9	20.6	1.38	0.003	5.50	0.00302	0.73
	15.4	29.4	1.91	0.003	8.14	0.00446	0.78
	14.75	46.5	3.16	0.003	11.75	0.00646	0.68
	15.4	15.4	1.00	0.004	5.45	0.00299	0.75
	14.6	15.5	0.94	0.002	3.14	0.00174	0.93
XDL5065, 70° F.	14.9	15.45	1.03	0.002	4.09	0.00131	0.64
	15.0	15.35	1.02	0.004	7.47	0.00240	0.59
	14.9	15.65	1.05	0.003	5.85	0.00187	0.60
	10.2	10.3	1.01	0.003	5.74	0.00184	0.61
	20.1	21.1	1.05	0.003	5.41	0.00173	0.55
	29.8	31.0	1.04	0.003	5.75	0.00184	0.59
	40.5	42.25	1.04	0.003	5.81	0.00186	0.60
	15.0	21.6	1.44	0.003	8.05	0.00258	0.60
	15.0	31.1	2.08	0.003	10.9	0.00349	0.56
	14.75	45.1	3.06	0.003	15.25	0.00489	0.53
XDH5005, 70° F.	8.64	25.2	2.92	0.003	10.49	0.00393	0.45

This coating is taken up completely by the paper moving with the velocity of the backing roll. Hence, the wet thickness, t, will be dependent on the ratio, R, of the casting and backing roll speeds,

$$t = t_1 R = kDR$$

To test this relationship the quantity t/DR was calculated and is shown in Table III. Average values of the coating constant k were determined for each mixture and are shown in Table V.

Table IV

Color	Backing-roll speed, f.p.m.	Casting-roll speed (critical), f.p.m.	Clearance, in.	Ratio, roll speeds
Gelatin	29.0	17.5	0.003	0.60
	12.5	7.4	0.003	0.59
Casein-clay	28.6	26.8	0.0015	0.94
	29.5	23.9	0.002	0.81
	28.8	22.7	0.003	0.79
	28.2	21.6	0.004	0.77
XDL5065	14.9	14.4	0.002	0.97
	14.7	10.8	0.003	0.73
	15.0	11.8	0.004	0.79
XL5426	14.6	12.5	0.002	0.86
	14.7	12.1	0.003	0.82
	15.4	8.4	0.004	0.55

Since $t_1 = kD$, the constant, k represents the ratio of the actual film thickness on the casting roll to the clearance between the casting and metering rolls. Factors which might be expected to affect the value of k would include the rheological properties and surface tension of the coating composition, the ratio of metering to casting-roll speed, and the head (depth) of liquid in the reservoir.

While the speeds of the rolls were varied fourfold, R threefold, and the clearance between metering roll and casting roll about two and one half fold, no definite trend in the value of k with any given factor could be discovered. For any one mixture the value of k remained relatively constant and the variation in k

Table V. Coating Constants (k) for Mixtures Studied

Coating mixture	Number of runs	k	Standard deviation
Gelatin-water	11	0.52	0.075
Casein-clay	17	0.49	0.035
XL5426	10	0.75	0.104
XDL5065	10	0.59	0.031
XDH5005	1	0.45	...

from mixture to mixture does not appear to tie in with the rheological differences between the five coating mixes. For the aqueous dispersions k is approximately 0.5, while the organosol gives a somewhat higher value and the vinyl solution XL5426 a 50% higher k, although the XL5426 is not as viscous as the organosol.

The hydrosol vinyl coating mixture should be commented upon. XDH 5005 was the only mixture which proved almost impossible to coat with the clearances and speeds utilized for the other colors, a uniform, even coating being impossible to attain. This may be due to the markedly thixotropic nature of this mixture since its apparent viscosity is not abnormal. In the one successful coating made, the coverage was of the same order of magnitude as that found for the other mixtures with the exception of XL5426.

Variation of the ratio of metering roll: casting roll speeds from 0.452 to 0.175 gave a variation in the value of k from 0.5 to 0.8 for a casein-clay color. While these experiments were only semiquantitative in nature, they were sufficient to indicate that this is an important variable.

Summarizing, it appears that coverage can be predicted with a fair degree of accuracy by the relation:

$$t = kDR$$

With the particular metering roll-casting roll speed ratio used (0.33) for aqueous dispersions k had a value of about 0.5. Below a certain critical value of R, in no case found to be greater than 1, the coating becomes nonuniform.

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Development of a Method for Treating Strawboard Wastes

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The previous work on the anaerobic digestion of strawboard wastes is discussed and the conclusions are given. The next step in studying the problem was the designing of a pilot plant. The necessary considerations that must be taken into account in designing, locating, and constructing a pilot plant are pointed out. In order to conserve digestion tank capacity it was decided to treat only to rotary-beater-washer wastes by the anaerobic process it being believed that the machine wastes would be about equal in waste concentration with the effluent from the anaerobic process. The anaerobic digestion process was started with digested sewage sludge. The details of starting the process are given. The available waste to be treated had a B.O.D. of 3560 p.p.m. as compared to a B.O.D. of 955 p.p.m. for the total waste used in the laboratory tests. The removal of total solids and B.O.D. obtained by primary sedimentation were 21.6 and 31.0% indicating quite effective treatment by sedimentation. The digestion tank was maintained at about 33°C. which is approximately the average temperature of the waste being treated. The B.O.D. reduction amounted to 0.0475 pound per cubic foot per day with the production of an effluent with a B.O.D. of about 135 p.p.m. The main difficulty in operation has been the bulking of the sludge. This resulted in a problem in sedimentation in the secondary settling tank. This difficulty was not believed to have been caused by the accumulation of solids because data indicated no increase in solids concentration in the digestion compartments even with no wasting of sludge. Laboratory tests have been conducted to determine the relative values of ferric chloride, aluminum sulphate, ferric sulphate and straw pulp as aids in filtering primary sludge. The indications are that all of these agents make the sludge filterable. On the basis of known methods estimates have been made of plant sizes in order to present a better idea of the plant that would be required should it be built today. It would seem that it would be advisable to continue studies to see if material reductions in treatment units and costs of operation cannot be made.

Two years ago a report was made at the Annual Meeting of the Technical Association of the Pulp and Paper Industry on the laboratory studies of the anaerobic digestion or treatment of settled strawboard waste. The conclusions drawn from the laboratory investigation were:

1. Settled strawboard waste with a B.O.D. of 600 to 800 p.p.m. when treated by the anaerobic process will result in an effluent with a B.O.D. of less than 75 p.p.m.
2. The digestion period necessary to obtain a satisfactory effluent is less than 2 days.
3. An effluent of less than 100 p.p.m. of B.O.D. can be produced with a 2-day detention time with the digestion tank held at 30°C.
4. To produce an effluent with 100 p.p.m. of B.O.D. at 25°C. requires almost 4 days detention. Marked reductions in B.O.D. can, however, be produced with shorter detention periods and still maintain good digesting conditions.
5. Practically no sludge is produced in the rapid digestion at the temperatures of 37, 30, and 25°C.
6. The primary sludge that would be produced in preparing the settled strawboard waste would have to be treated with chemicals and vacuum filtered or digested in separate digestion tanks and then filtered if present known methods of disposal are to be used.

The conclusions did not state that the next step in the process of development of the anaerobic process was to build and operate a pilot plant but it is a well-known fact that few investigators have the courage or shortsightedness to build full scale plants based on studies made in bottles in a laboratory. It would often be much easier to decide on the size of a full scale plant than it would be to come to a conclusion on the suitable size for a pilot plant.

The laboratory studies had pointed out the advantage of increased temperature in accelerating the proc-

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ess. With present day costs of heating it was out of the question to try to raise the temperature of the entire mill waste. In a 100-ton mill the flow of waste waters amounts to approximately 2 million gallons. A few temperature readings of the wastes from the cooker-beater-washer operations and the paper machines confirmed the belief that considerable increase in temperature could be gained if the machine waters could be excluded from the digestion process. Previous studies (*1*) on waste characteristics showed that the machine waters usually have about the same B.O.D. as the effluents expected from the anaerobic process. It seemed advisable under these conditions to try to treat anaerobically only the cooker-beater-washer wastes. If these more concentrated wastes could be treated there could be a considerable reduction in disposal plant size and elevation of temperature of the waste to be treated would be accomplished. Changing concentrations of waste when going from the laboratory investigation to the pilot-plant stage might have been considered a very questionable thing to do if it had not been known that in the anaerobic treatment of other wastes (particularly yeast wastes) the concentrations are higher than those expected in the more concentrated strawboard waste.

With these proposed changes, steps were taken to design a pilot plant to be located at the No. 2 mill of the Terre Haute Paper Co.

The size of the pilot plant is usually governed by a great many factors but there are two criteria that must be satisfied. These desires are: (1) to build the pilot plant as large as possible, and (2) to build the pilot plant as economically as possible.

Some designers say that pilot plants should be built to handle 10% of the total waste to be eventually treated. Earlier measurements had indicated that the rotary-beater-washer waste volume amounted to about one half of the total waste flow. On the basis of the 2 million gallon figure for total waste flow previously given there would be approximately 1 million gallons for treatment should the anaerobic process prove to be suitable. On the 10% basis of design the daily flow to be handled by the pilot plant would be 100,000 gallons and with an anticipated 2-day detention time a digestion tank of 200,000 gallons capacity would be needed. It did not take long to decide that a pilot plant of such a size could not be built with available funds, and it was thought extremely doubtful whether a pilot plant of such a size was really necessary. If this basis of design gave a pilot plant of unreasonable size then the question was what basis of design can be used. It was finally concluded that the minimum flow through the digestion tank should never be much less than 5 g.p.m. if any degree of accuracy was to be obtained in the measurement of flows.

The pilot plant location selected was a level plot of ground immediately adjacent to the beater room of the mill. The pump necessary for discharging waste to the pilot plant was located so that suction could be taken from a channel carrying rotary-beater-washer waste or machine-water waste or a combination of both wastes. A manhole on the main sewer from the plant was within easy reach of the contemplated location of the pilot plant so that there was no problem in the disposal of the effluent or other drainage. Power and steam were readily available. Complete facilities for chemical tests were at hand in the paper mill laboratory.

The general layout of the plant was to be a primary sedimentation tank, sludge digestion tank, secondary settling tank, waste sludge storage tank, and pumping equipment for returning the sludge from the secondary tank to the digestion tank.

The primary tank was to be a very important part of the pilot plant because the waste was to have primary sedimentation prior to treatment in the digestion tank. It was also extremely important to learn the degree of treatment obtainable with straight sedimentation. Previous primary treatment tests had been made in a precipitator type tank.

SYSTEM OPERATION

Consideration was given to the type of construction to be used in building the pilot plant. In constructing a temporary structure wood is always given first consideration because of cost and usual ease of fabrication. The pilot plant was to be built above ground and any leaks would have been very objectionable because of the particular location. When concrete is used there is always the problem of removal after the tests have been completed. Upon investigation a steel tank was found that was of suitable size and shape and it was altered to give units of the sizes shown in Table I.

Table I. Pilot Plant Tank Sizes

Unit	*Width, ft.*	*Length, ft.*	*Depth, ft.*	*Total volume* — *Cubic feet*	*Gallons*
Primary tank	2	10	11.5	159.5	1,193
Digestion tank	10	30.33	11.0	2980	22,300
Secondary tank	2	10	11.0	149.6	1,120

The plant design flow was taken as 15.5 g.p.m. or 22,300 gallons per day. The detention time in the primary tank at design flow, assuming that the sludge would be 4% of waste flow and stored for 12 hours, would be 48 minutes. The detention time in the digestion compartment at design flow would be 1 day. The period of retention in the secondary tank at the design flow would be 47 minutes.

A particularly novel design of weirs was made which allows control of flow to the primary tank without the necessity of adjusting the discharge of the pump supplying the waste and which also permits taking only a portion of the primary effluent into the digestion tank. The effluent from the secondary tank is discharged over a V-notched weir with means of recording the head on this weir. A sludge wheel was designed to return the sludge from the secondary tank to the digestion tank. Such a device was believed to be advantageous because the rate of pumpage could be varied by changing drive pulley drives or sizes of buckets. A pump handling such small quantities of material would, it was thought, be too difficult to keep in operation.

The primary and secondary settling tanks were hopper bottomed for sludge collection. The digestion tank was divided into three compartments each having a mechanism similar to that used in chemical flocculation units. The purpose of this equipment was to mix the incoming raw waste with the seed sludge. The compartments of the digestion tank were each covered with two floating covers for gas collection.

Arrangements were made with the city of Marion, Ind. to deliver 1500 gallons of digested sewage sludge to serve as seed in starting the digestion tank. The pilot plant tanks were then filled with waste, taking 5 days to complete the operation so that the unit would not be overloaded with organic matter. The plant was then allowed to idle for 13 days to make sure that there was no chance for overloading.

Characteristics of the Waste

The rotary-beater-washer waste was found to have a temperature of between 29 and 33 °C. The waste for the pilot plant was found to be much stronger than had been anticipated. A comparison of the total mill waste with the rotary-beater-washer waste is made in Table II.

Primary Sedimentation

The extreme difficulty in obtaining check results on suspended solids because of the very fine particles in the waste has prompted the discontinuation of the suspended solids test as a method of showing waste strength or efficiency of removals. A comparison of the operating results of the present pilot plant primary settling tank and precipitator-type tank operating on the less concentrated waste, is given in Table III.

It is apparent that primary sedimentation is quite effective in removing solids and B.O.D.

OPERATION OF DIGESTION TANK

The pilot plant was started in the fall of the year and upon arrival of the first cool weather it was apparent that the metal tank would radiate so much heat that the temperature of the raw waste could not be maintained. Loss of heat was stopped by building a wooden tar papered housing around the pilot plant and then banking it with straw and installing two unit heaters to make up any loss of heat.

Typical of the operation of the digestion unit is shown by the data collected during the period Jan. 16 to Feb. 7, 1948. During this period the pounds of B.O.D. removed per cubic foot of tank amounted to 0.0475. This removal amounted to 95.5% of the B.O.D. added. The effluent produced during this period had a B.O.D. of 136 p.p.m. The operating temperature during the same period was 33 °C.

The sludge wheel that was thought to be such an excellent idea for returning sludge did not stand the wear that was experienced. It was necessary to replace the wheel with a piston pump. The pump was built in the shop of the Terre Haute Paper Co. and it has served for several months with no particular difficulty.

Table II. Comparison of Total Mill Waste and Rotary-Beater-Washer Waste

Analysis	*Total mill waste*	*Rotary-beater-washer waste*
Total solids, p.p.m.	3691	8280
Volatile total solids, p.p.m.	2449	5840
B.O.D., 5-day p.p.m.	955	3560
Volatile per cent of total solids	66.4	70.6
B.O.D., pounds per pound of total solids	0.26	0.43
B.O.D., pounds per pound of volatile total solids	0.39	0.61

Table III. Comparison of Sedimentation Tanks

Data	*Precipitator pilot plant No. 1*	*Sedimentation tank pilot plant No. 2*[a]
Total solids in raw waste, p.p.m.	3701	7940
Total solids in primary effluent p.p.m.	3051	6402
Removal of total solids, %	17.5	19.1
B.O.D. in raw waste, p.p.m.	954	3620
B.O.D. in primary effluent, p.p.m.	823	2780
Removal of B.O.D., %	13.8	30.2
Concentration of sludge removed, %	0.68	4.52
Overflow rate, gal./sq. ft./day	1915	448

[a] Period Dec. 1, 1948 to Feb. 21, 1949.

The chains used to drive the mixing mechanisms were old when first put into use in the pilot plant and they have given trouble from time to time. It is believed that well fitted, new chains would give no particular difficulty if they were used in a full scale plant.

There is a difficulty that has arisen that has not been overcome. After several months of operation it was observed that at times it was impossible to remove the sludge fast enough from the secondary tank. It was first thought that there was some difficulty with the returning mechanism but it was finally concluded that the sludge from the digester would not settle. In laboratory tests on settling the sludge behaved much as a bulking activated sludge. This difficulty has not been overcome as yet. Changes in design of the pilot plant recently made will permit the addition of raw waste to the digestion tank. It is hoped that the extra weight of the solids in the raw waste will suffice to weight the sludge so that when discharged to the secondary settler the sludge will settle out quickly and effectively. Even during the period of bulking it was apparent that the effluent being produced showed a reasonable B.O.D. reduction.

The effluent produced in the pilot plant was not as of high quality as was the effluent produced with the laboratory set-up. It must be borne in mind that waste handled in the pilot plant was almost four times the strength of the waste used in the laboratory experiments.

During one period the digestion tank was overloaded by mistake and the recovery period was followed very closely with the volatile acids determination. At the high the volatile acids concentration was 1800 p.p.m. and in 2 weeks they were down to below 300 p.p.m. The volatile acids test is a most useful tool in following the operation of the process.

The total solids in the digestion tank have been maintained at about 1%.

No sludge was wasted from the digestion tanks until the difficulty with bulking was experienced. If there had been much of an increase in sludge in the digestion tanks there would have been an increase in concentration.

SLUDGE DISPOSAL

When mill superintendents were asked about their losses there was some difference in the quantities reported but the usual answer was about 36%. An actual calculation of losses based on the analysis of waste from the Terre Haute Paper Co. Mill No. 2 revealed that the loss amounted to 29.26%.

The removal of total and suspended solids by the precipitator averaged 17.5 and 56.1%. The removal obtained in the primary sedimentation tank of pilot plant No. 2 was 21.6% for total solids. It must be kept in mind that this was a more concentrated waste than was used for the tests with the precipitator. Based on the average of daily results for a 5-month period there were 55.97 tons of total solids and 22.3 tons of suspended solids discharged from the mill per day.

If the removals obtained with the present pilot plant are applied to the data just given the amount of sludge solids as primary sludge to be disposed of per day would amount to 12.1 tons. The usual concentration of sludge from the primary tanks is about 4.52%. Using this percentage and the 12.1 tons in a computation to find the gallons of sludge produced from a 100-ton mill the answer comes out to be 64,200 gallons.

Eldridge (*2*) found a number of years ago that strawboard waste sludge could be digested. It would seem, however, that the usual long periods required for solids decomposition would necessitate large digestion tanks.

A laboratory investigation of various chemicals and materials was made to see how practical it might be to dewater the primary sludge on vacuum filters. Ferric chloride, aluminum sulphate, ferric sulphate, and prepared straw pulp were used as conditioning agents. The sludge can be filtered with any of these agents. All of them will produce filter cakes with moisture contents of from 69 to 78%. The amount of ferric chloride ($FeCl_3$) required was 12 to 15% of the dry weight of the solids filtered. The amount of aluminum sulphate ($Al_2(SO_4)_3$) was between 6 and 7% of the dry solids filtered. The amount of ferric sulphate ($Fe_2(SO_4)_3$) required for suitable filtration was 18.6% of the dry solids filtered.

The concentration of the sludge filtered was about 5%. This is a concentration higher than that easily obtained from primary sedimentation. Further studies will be necessary to determine whether the sludge of lower concentration will require substantial increases in the quantity of chemical required per unit weight of dry solids.

Based on current car load quotations for these chemicals the cost for chemicals alone per ton of dry solids would be; ferric chloride $17.25, aluminum sulphate $4.08, ferric sulphate $5.30.

A number of years ago there was some experimental work done on the use of paper pulp as an aid in the vacuum filtration of sewage sludge. The sewage sludge could be filtered with the pulp but the process was not developed because of the fluctuation in the market price of paper. Realizing that there is no place where pulp can be procured as cheaply as in a paper mill it was decided to try filtering primary sludge with straw stock. The indication from one test indicated that it would take 165% by weight of pulp to filter the primary sludge. Multiplying this percentage times the 22.3 tons of waste solids from a 100-ton mill gives 36.8 tons of pulp required for filtering the 22.3 tons of primary sludge solids. This would be out of the question unless the sludge and pulp could be processed into paper without the loss of efficiency in manufacturing or quality of product. There is one mill now operating on this basis but the quantity of straw processed is only a small portion of the total tonnage of paper produced.

It is realized that it is extremely dangerous to make statements regarding plant-sized units when the experimental data offered are not conclusive. It is very difficult, however, to visualize or interpret findings from research unless they are expressed in a magnitude comparable with known existing units. An attempt will be made to give some idea of the size of plant needed to treat the waste from a 100-ton mill.

A 100-ton mill will discharge about $3^1/_2$ million gallons per day. To give this amount of waste primary treatment of the degree of treatment indicated (21.6% removal of total solids) would require a detention time of about 1 hour. This then would mean that primary tanks with a volume of 146,000 gallons would have to be provided. The 64,200 gallons of sludge produced per day would have to be disposed of by lagooning or be filtered with disposal of 48.4 tons of filter cake of 75% moisture content. To filter 64,200 gallons of sludge per day it would be necessary to provide two standard vacuum filters with drums 11.5 feet in diameter and 14 feet long. The quantity of filter alum required per day would be at least 1 ton.

Calculations for volume of rotary-beater-washer waste made on the basis of 3.5 m.g.d. for total waste and the B.O.D. relations shown on Table II indicate that there would be approximately 865,000 gallons of rotary-beater-washer waste to dispose of per day. Duplicating the results obtained in the pilot plant would require digestion tanks with a volume of 520,000 cubic feet (4,320,000 gallons). Allowing for a return of sludge from the secondary settling tanks amounting to 50% it would be necessary to provide a settling tank volume amounting to 54,000 gallons if a 1-hour detention time were provided.

CONCLUSIONS

1. It is probable that a strawboard waste treatment plant using the anaerobic process could produce an effluent with a B.O.D. of 150 p.p.m. or less from rotary-beater-washer waste waters.

2. Primary treatment of strawboard waste will remove 19% of the total solids and 30% of the B.O.D.

3. Sludge from primary sedimentation can be filtered on vacuum filters.

4. It is believed that the cost of sludge filtration by presently known methods will be very high.

5. Because of difficulties in operation of the anaerobic process further tests must be completed before a full scale plant is constructed.

LITERATURE CITED

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Some Factors Affecting Slime Formation and Freeness in Boardmill Stock

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Recirculation of white water is a common practice in most paperboard mills as a method of pollution abatement and of conservation of fiber, filler, and water. Slime, one of the major nuisances resulting from this recirculation, causes a decrease in the drainage rate (freeness) of the stock which is measured by a laboratory handsheet machine. Slime forms to some extent when white water is recirculated without pH control and at a temperature range of 20 to 37°C. The extent of slime growth and its optimum growth temperature will vary somewhat from one boardmill to the next. A pH of 4.5 or a temperature of 50°C. was found to prevent slime growth. Replacement of the nonsettleable portion of white water with fresh water, before recirculation, inhibits slime growth. Slime formation is greatly stimulated by casein, slightly stimulated by starch, and not affected at all by rosin.

Recirculation of paperboard white water is frequently considered objectionable because of slime formation and the subsequent fouling of fibers or even slowing down of the machines. Slime is a mass of bacterial excretion products enmeshed in and around partially decomposed pulp and fungi. Buckman (*1*) likens this slime mass to concrete, in which the capsular bacterial excretions act as the cement binder and the fungi and pulp fibers act as the retaining structure. It can easily be visualized how such a slimy mass can slow down the drainage rate of a stock on a wire of a paper machine. The drainage rate is a characteristic of boardmill stock best described by the term "freeness." An increase in the drainage time corresponds to a similar decrease in freeness of the stock and vice versa. Since slime causes a decrease in freeness, and the latter is measured best by the drainage time, slime also can be measured by the drainage rate of a stock. It is realized that the type of furnish and the degree of fibrillation can also affect the freeness. Keeping various conditions constant, an attempt has been made to use freeness as a direct measurement of slime formation. Further, an investigation was made concerning some of the factors affecting freeness of boardmill stock during the recirculation of white water.

METHODS AND MATERIALS

Since this paper deals with one of the main problems encountered in the recirculation of white-water wastes in the chipboard mill, it is appropriate that an analysis of the white water be given (Table I). This analysis closely represents the physical character of the many white-water samples collected. Generally, all samples were collected on the same day of the week at the same mill making essentially the same paperboard product. However, variations exist in boardmill white waters even within the same mill.

"Standard Methods for the Examination of Water and Sewage" (*2*) was used in all analyses made on the white water unless otherwise specified.

All tests were made at room temperature, except when the effect of various temperatures was studied.

Apparatus

To determine the magnitude of slime formation an apparatus was designed and built for measuring any changes in the drainage time (freeness) of the stock. The apparatus, called the "Laboratory Handsheet Machine," is an adaptation of the more or less "standard" handsheet mold. The machine used is illustrated in Fig. 1. It consists of a wire screen (40 mesh) supported by a larger screen and rubber gaskets between the faces of two pipe flanges. Above the screen and threaded into the upper flange is a 4-inch short length of pipe. This pipe serves to hold the white water until it drains through the screen. Underneath the screen is a short length of 4-inch pipe reduced to a 1-inch valve. This pipe allows for the collection of the filtrate. Directly underneath the flanged joint and behind the box, a $^3/_4$-inch line is tapped and threaded which leads to a 2-inch length of pipe connected directly at one end to the suction end of a small vacuum pump. A gate valve in this $^3/_4$-inch line controls the instantaneous vacuum applied to the stock underneath the screen. The 2-inch vacuum storage line is fitted

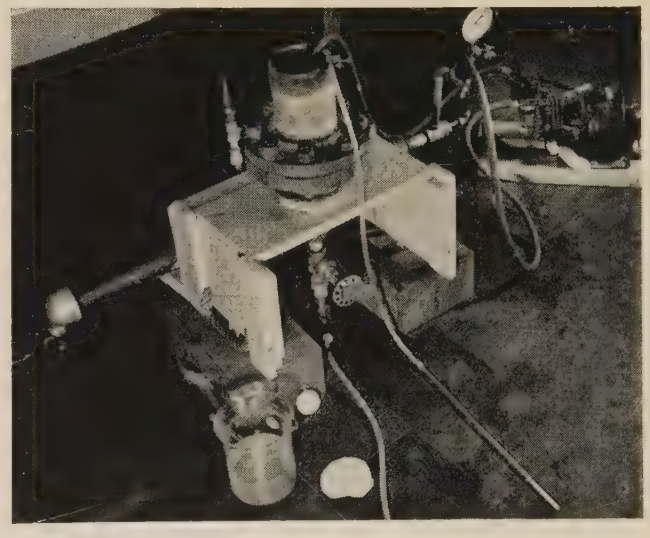

Fig. 1. Overlooking front view of laboratory handsheet machine

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at one end with a drain cock and at the other end with a vacuum gage and bleed-off control petcock as well as the suction connection to the vacuum pump.

Operation of Machine. After the vacuum pump is started, the suction line is connected to the 3/4-inch valve just beneath the screen and a 1-liter sample of the white water to be tested is poured onto the screen. Immediately the suction valve is opened in the 3/4-inch line and the bleed-off cock manipulated to show a reading of 2 inches on the gage. When the white water is completely drained through the screen the fibrous mat left changes to a lighter shade of gray almost instantaneously and appears dry. The sharp change in color was used as one of the indices for the completion of filtration. The stopwatch is snapped off at this point and the drainage time recorded (stopwatch). The vacuum is disconnected and the 1-inch valve opened, releasing the filtrate to be used over. The discharge end of the vacuum pump is then connected to the 3/4-inch hose also leading directly beneath the screen. The fibrous mat is then blown loose from underneath the screen and removed by hand from above.

Table I. Analysis of White-Water Waste from Chipboard Mill

pH	7.0
Alkalinity as $CaCO_3$, p.p.m.	118
Suspended solids, p.p.m.	840
Fixed suspended solids, %	11.9
Volatile suspended solids, %	88.1
Total solids, p.p.m.	2180
Ash, %	19.3
Volatile total solids, %	80.7
Dissolved solids, per cent of total	61.5

To clean the screen thoroughly for the next test, clear water is forced upward into the filtrate line through the screen, thus lifting out any entrained fibers. Compressed air is also introduced above the screen into the upcoming water for agitation. When the water has reached the 1-liter mark above the screen, it is siphoned off into the drain. Then the clear water beneath the screen is released through the filtrate line. After the filtrate valve is closed the apparatus is ready to be used again. This method indicating "freeness," a physical phenomenon, can be used to measure accurately the extent of slime formation, assumed to be a biological growth.

EXPERIMENTAL RESULTS

Simple Mechanical Recirculation

For the purpose of these experiments the term "recirculation" signifies the incubation of white water plus the fiber sheet and the subsequent returning of the mixture to the screen. The term "adjustment of consistency" signifies the appropriate amount of dried paperboard added to make a specific concentration of fibrous material. To determine whether the physical character of the white water is altered by constant recirculation (picking up new fiber stock and passing through the machine screen) a liter of white water adjusted to 0.25% consistency, at a pH of 4.5 and a temperature of 38°C. was recirculated through the machine seven times within eight hours, with the liquid adjusted previous to each passage. The adjustment of the consistency, pH, and temperature of the recirculated white water was intended to simulate practice followed in several boardmills.

Table II. Continuous Recirculation of White Water

Recirculations, No.	*Drainage time, sec.*	*Suspended solids in the recirculated white water, p.p.m.*
1	51	70
2	66	30
3	69	50
4	60	50
5	66	50
6	60	46
7	55	60

The results (Table II) show that white water, which was kept circulating in a clean system at constant temperature and pH, does not slime up and cause a decrease of freeness of the material on the screen, indicating that the mechanical act of continual recirculation of the white water is not responsible for a change in the character of the water causing it to decrease in freeness. It is possible that the increased dissolved solids after recirculation would become susceptible to bacterial action if the white water were allowed to stand for a considerable time or to come in contact with "dead spots" in the lines during recirculation.

Nature of Solids Build up in Recirculated White Water

To simulate actual conditions in a boardmill 2.5 grams of dried paperboard stock was added to a liter of distilled water, beaten in a Waring Blender at 65°C., adjusted to a pH of 4.5 with alum, and poured onto the screen of the handsheet machine at 100°F. The "freeness" of the stock was measured. The same amount of fiber was added to the filtrate before beating and adjustment of the temperature and pH was just as before. This procedure was repeated twice a day for 3 days with three samples of artificial white water. The first sample contained only distilled water and fiber, and the other two samples were seeded with fresh sewage and refortified with N and P of various amounts (*3*).

Table III. Build up of Solids in Recirculated White Water

Sample No.	*Incubation at 37.5°C., hr.*	*Total solids, p.p.m.*	*Suspended solids, p.p.m.*	*Dissolved solids, p.p.m.*	*Volatile dissolved solids, p.p.m.*	*Freeness of white water, sec.*
1	0	187	54	133	58	49
	18	235	60	175	74	54
	36	313	80	233	108	41
	54	348	57	291	127	43
	72	299	59	240	107	40
2	0	186	61	125	65	43
	18	245	53	192	84	49
	36	271	58	213	84	42
	54	243	53	190	94	42
	72	333	83	250	124	56
3	0	130	55	75	28	42
	18	128	81	47	74	56
	36	206	68	138	74	54
	54	259	57	202	75	54
	72	262	57	205	114	55

From the results (Table III) it appears that the buildup of solids in laboratory recirculated artificial white water was caused entirely by an increase in dis-

solved material or colloidal matter which passes the asbestos mat of a gooch crucible. However, the build up occurred in all samples, but freeness decreased only in samples seeded with sewage bacteria. It appears that the decrease in freeness of the recirculated white water is caused by the excess dissolved matter being utilized as bacterial food and not to the dissolved material as such.

Effect of Initial pH Adjustment of White Water

Twenty-four liter samples of natural boardmill white water were incubated at room temperature (21°C.) for one to five days. Twelve of the samples were agitated intermittently and the others allowed to stand quiescently. The 24 samples were divided into three sets immediately after being poured; the pH values adjusted to 4, 6, and 8, respectively, before incubation as described above. It was found that regardless of the initial pH of the white water (from 4 to 8) and despite the physical treatment of the water (intermittent stirring or quiescent conditions) the pH values tend to come to an equilibrium range of between 6.2 and 7.2. When the material is stored under quiescent conditions the time required to attain equilibrium is longer.

Effect of Controlled pH on the Freeness of Recirculated White Water Waste

Certain changes take place in the recirculated white waters, believed to be biological, which affect the pH of the waters. Experiments were conducted to determine in which pH range slime forms and to what extent; and conversely in which pH range or ranges slime formation can be most effectively controlled.

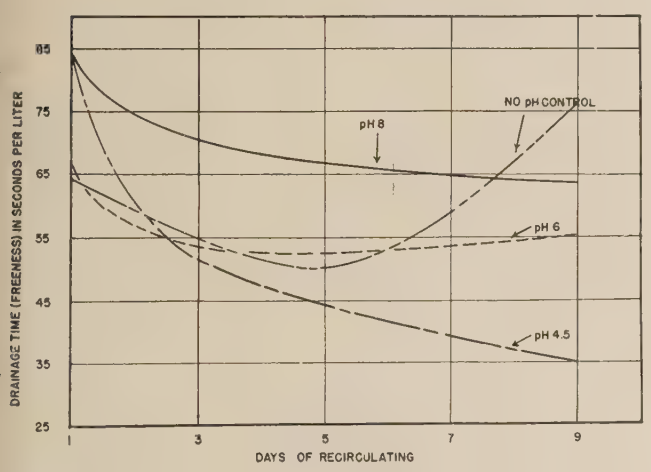

Fig. 2. Effect of controlled pH on slime formation and freeness in boardmill stock

The pH values of six 1-liter representative natural boardmill samples of white water were altered as follows:

1. A 20% slurry of lime was added until the pH rose to 8.
2. A 40% solution of caustic soda was added until the pH rose to 8.
3. Alum solution was added until the pH dropped to 6.0.
4. Alum solution was added until the pH dropped to 4.5.
5. Concentrated H_2SO_4 was added until the pH dropped to 4.5.
6. No chemicals were added (control).

The samples were exposed to slow, intermittent, mechanical stirring for one day. A total of 2.5 grams of previously pulped and dried paperboard stock was then added to each sample, after which all samples were thoroughly mixed and poured onto the screen of the laboratory handsheet machine. The time of filtration was noted, and the paper retained on the screen was immediately recombined with the filtrate. At this point in the procedure each day pH adjustments were made on each sample. No more stock was added to the samples than was removed, so that the solids remained constant during the run.

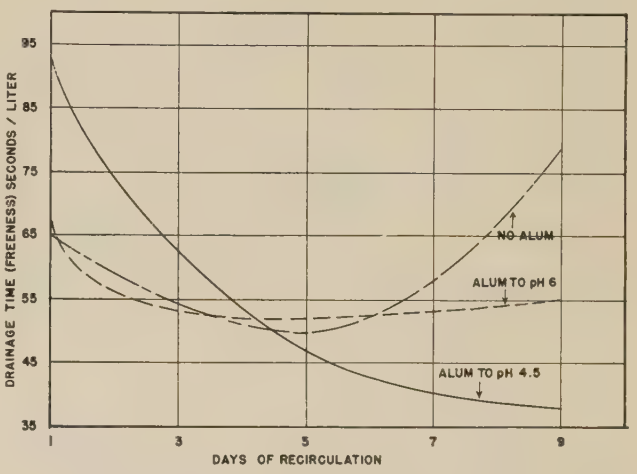

Fig. 3. Effect of alum as a pH regulator on slime formation and freeness of boardmill stock

The results shown in Fig. 2 indicate that the freeness of the recirculated white-water stock was a time reaction under controlled pH conditions. When the pH was controlled with alum or sulphuric acid to 4.5 no slime formation resulted as shown by the constant increase in freeness or decrease in drainage time in the recirculated white water. With no pH control, slime formation took place after 5 days and the freeness of the white water decreased; the sample had a foul and fishy odor, whereas the samples controlled to pH values lower than 6 had no odor. By controlling the pH at 8 with lime or caustic soda the increase in freeness was slight and showed tendencies to slime up at times. At the end of nine days a liter of "uncontrolled pH" stock took 137% longer to drain than the sample of white water controlled at pH 4.5 with sulphuric acid. The drainage time was 92% longer when no pH control was used than when the white-water sample was controlled to pH 4.5 with alum. Figure 3 shows the effect of alum as a pH regulator on the slime formation and freeness of the recirculated stock.

Effect of Temperature of Environment on "Sliming Up" of Recirculated White Waters

To determine the effect of temperature on sliming, previously pulped and dried paperboard stock was added to white water and incubated for 4 hours at 10, 20, 30, 37.5, or 50°C., until a constant temperature was reached. Then the samples were each separately poured onto the screen of the laboratory handsheet machine and their drainage times (freenesses) recorded. The mats from the screen were then recombined with

their respective filtrates and incubated again at their previous temperatures.

The results of the freenesses during the run, shown graphically in Fig. 4, indicate that with waste 1 the freeness of recirculated white water decreased with

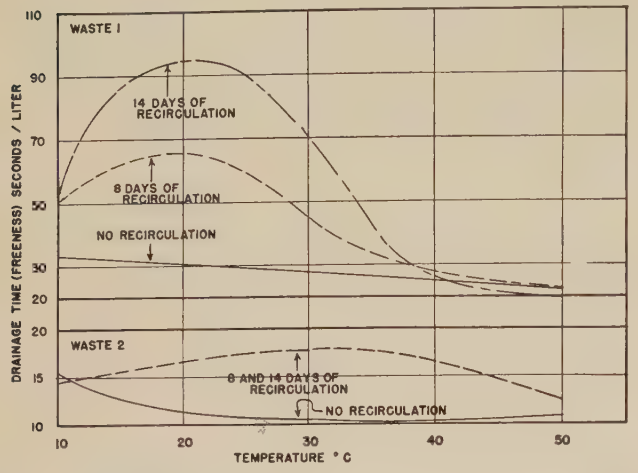

Fig. 4. Effect of controlled temperature euvironment on the slime formation and freeness change of recirculated boardmill white water

time when at 10, 20, and 30°C., but at 37.5 and 50°C. no decrease took place. The optimum slime conditions existed at 20°C., and slime formation decreased above 20°C. until at about 40°C. none occurred. A temperature of 20°C. appeared to be ideal for slime growth, resulting in a maximum decrease in freeness of 237% after 14 days. At 30°C. a decrease in freeness of 163% took place after 14 days, and a 49% decrease in freeness occurred at 10°C. after eight days. An initial increase in freeness of the waste of 36% occurred when the temperature was raised from 10 to 50°C.

With waste 2, the optimum slime growth occurred between 25 and 37.5°C., with the maximum at 30°C., resulting in a maximum decrease in freeness of 67%. White waters incubated at 50°C. filtered faster at all times during the 13 days—including the maximum slime growth period—than white water incubated at lower temperatures. An initial increase in freeness of about 33% was obtained by increasing the temperature of the recirculating stock from 10 to 50°C.

The results indicated that the extent and character of the slime growth may vary from place to place and the effect of temperature may be more pronounced at one place than another.

Effect of $HgCl_2$ on the "Sliming Up" of Recirculated White Water

To prove or disprove that bacterial action results in slime formation and that the slime which results can be expressed in freeness and measured by means of the laboratory handsheet machine, two different wastes were studied. In both cases 30 p.p.m. of $HgCl_2$ and about 3 grams of paperboard stock per liter were added to the white water which was incubated at 20°C. for several days. Waste A was incubated at 30°C. after 3 days to induce faster slime formation. The results of pH and freeness changes during the experimental periods are shown graphically in Figs. 5 and 6. With waste A, $HgCl_2$ caused a 28% increase in freeness of the recirculated white water after 8 days. Bacterial action was sufficiently inhibited to prevent changes in pH during the experiment. Without $HgCl_2$ addition, a 23.6% decrease in freeness occurred after 7 days. Bacterial action in this sample accounted for the characteristic pH curve which slowly rose above 6 where slime began to form. The waste to which $HgCl_2$ was added showed an increase in freeness of 22.2% after recirculation for 13 days as compared to 39.3% decrease in freeness with no $HgCl_2$ added. The results with the second boardmill white water waste show that a biologically uncontrolled, recirculated white water decreases in freeness (or increases in drainage time) after about a week of recirculation at relatively constant temperature (20 to 30°C.). The quantity of slime formed varied between the boardmill white water wastes. Boardmill waste B's white water took 100% longer to drain after 10 days of recirculation with no biological control than the same waste with $HgCl_2$ added to inhibit bacterial action.

Effect of Controlled Environment on Slime Formation as Measured by Freeness Change as Well as the Total Bacterial Count

It has been shown indirectly that slime formed in recirculated white waters was caused by bacterial action. An attempt was made to correlate directly the increase in slime formation (decrease in freeness) with a change in total bacterial population. In previous investigations recirculated white waters controlled to a pH of 4.5 or a temperature of 50°C. did not slime up as measured by an increased drainage time. Now, the effect of these same environmental controls on the bacterial population was determined.

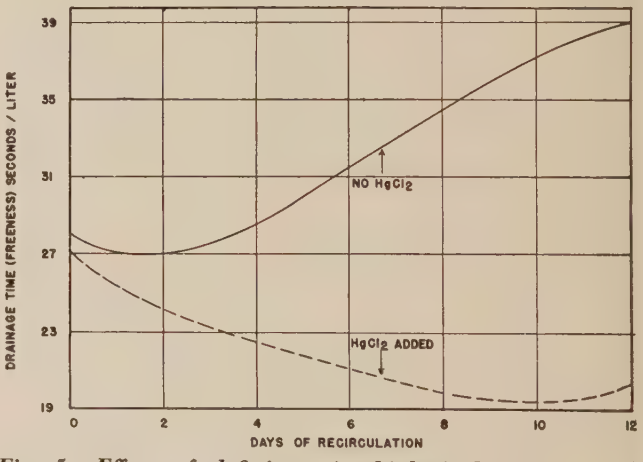

Fig. 5. Effect of definite microbiological control with $HgCl_2$ upon slime formation and freeness change in recirculated boardmill white water

Three liters of natural white water were blended with 9 grams of previously pulped and dried paper stock. Homogeneous, 1-liter samples were adjusted in the following manner:

1. No adjustment and incubated at 20°C.
2. No adjustment and incubated at 50°C.
3. Adjusted to a pH of 4.5 with alum and incubated at 20°C.

After adjustments had been made and the samples had come to the desired temperature, they were poured

onto the screen of the laboratory handsheet machine and their drainage times and pH values were recorded. The duration of the experiment was 17 days. Total bacterial counts were made at various times during the experiment. The agar plates were incubated at 20°C. for 2 days and then counted.

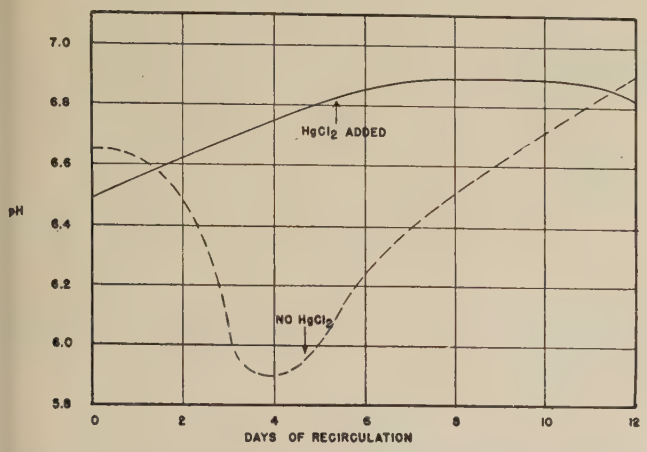

Fig. 6. Effect of definite microbiological control with $HgCl_2$ upon pH of recirculated boardmill white water

The drainage time increased 79% when the white water was recirculated daily and incubated at 20°C. for 17 days (Fig. 7). The drainage time of the water decreased about 16% when the temperature was raised from 20 to 50°C. No increase in drainage time (decrease in freeness) of white water incubated at 50°C. and recirculated daily occurred even after 17 days. The recirculated and 20°C. incubated white water controlled to a pH of 4.5 with alum showed a decrease in drainage time of about 17.5% at the end of 4 days and stayed at this level without showing a tendency to slime up for 17 days.

In unadjusted and 20° incubated white water the pH dropped initially and then rose slowly to 6.7 as slime started forming (Fig. 8). At 50°C. the pH was nearly constant for 5 days, at which point it rose slowly to 7.0.

In unadjusted and 20°C. incubated white water the total bacterial numbers decreased from 6,200,000 per ml. to about 100,000 to 300,000 per ml. at the end of 10 days of recirculation (Fig. 9). However, this decrease was at a slower rate and resulted in higher numbers than when controlled at 50°C. or pH 4.5. At 50°C. the numbers dropped from 6,200,000 to about 1000 to 2000 per ml. during the first 3 days and remained at this low level during the 17 days of the experiment. Similarly, at a pH of 4.5 the numbers were also reduced to between 2000 and 4000 per ml. at the end of eight days and also never recovered.

Other Factors Influencing Slime Growth

The fact that some samples of recirculated white water slimed up noticeably while others showed no slime formation led to a study of other factors which may be of importance. Obviously there are substances present in white waters which create favorable conditions for slime growth. It is possible that increased concentrations of food for organisms play an important role in growth stimulation and even change the physical characteristics of the white water to such an extent that the growth rate is affected. In this respect it is necessary to know in which physical fraction of the white water the slime originates, and to determine its rate of formation under various conditions.

Six 1-liter samples of a representative white water from a north Jersey paperboard mill were altered as follows:

1. No change (control).
2. Supernatant removed after settling and replaced by distilled water.
3. Removal of settleable solids by centrifuging and replacement with dried paperboard stock.
4. Addition of soluble starch (100 p.p.m.) to white water at start (each day freeness test was made).
5. Same as (4) with 100 p.p.m. of casein.
6. Same as (4) with 100 p.p.m. of rosin.

The results are shown in Table IV.

The "control" required 153% more time to drain after 18 days of incubation and recirculation at 20°C. than at the beginning of the test. The freeness appeared to decrease directly as a straight-line function of the length of time of incubation and recirculation. The sample which had its supernatant removed after settling and replaced by distilled water did not slime up. It appears that there was something in the removed supernatant that was vital to slime formation. The sample which had its original settleable fiber removed and replaced by fresh dried paperboard, slimed up 79.5% (as measured by the increase in drainage time). This shows that normal slime growth was depressed about 50% by the removal of the settleable solids.

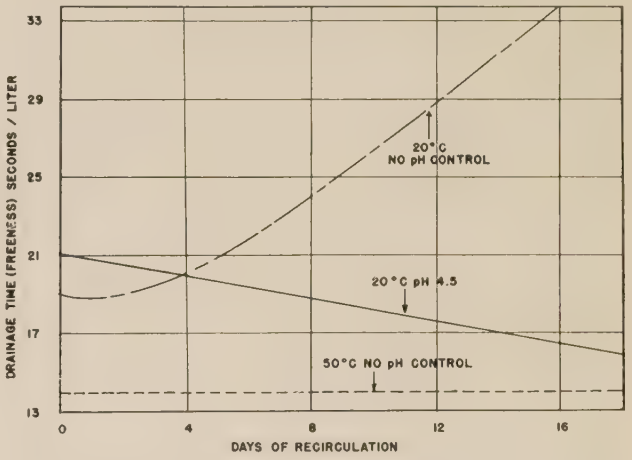

Fig. 7. Effect of controlled temperature (50 and 20°C. or controlled pH (4.5) on the slime growth and freeness of the recirculated boardmill white water

The sample to which was added daily 100 p.p.m. of soluble starch showed a 184% increase in drainage time. This indicates that a readily available source of carbohydrate food aids in slime formation causing 31% more slime growth. Addition of 100 p.p.m. of casein each time the white water was tested for freeness resulted in 518% sliming after 18 days. It is apparent that casein stimulated slime growth to a considerable extent. This means that the slime forming microorganisms present in the natural white water are sufficiently abundant to cause difficulties and that the

source of carbohydrate food for these microorganisms was present in ample amounts to promote slime growth, but that the slime growth was limited by the amount of available nitrogenous food in the water. After casein was added, a period of acclimatization, during which the breakdown of the complex protein undoubtedly occurred, was followed by vigorous slime growth and slime growth at the end of 9 days even though the temperature was ideal for the slime-forming microorganisms. An "uncontrolled pH" sample of this same recirculated white water took 137% longer to drain through the wire of the laboratory handsheet machine. Sulphuric acid proved slightly better as a pH regulator than alum, but both worked well. This

Table IV. Factors Affecting Rate and Extent of Slime Build up

Sample	*Key*	*Freeness (drainage time) after the following number of days of recirculation and incubation, sec.* 0	1	2	3	6	7	8	10	13	18
1	Control	17	19	22	21	24	25	24	31	31	43
2	Replaced supernatant	16	17	19	17	19	19	19	20	19	19
3	Replaced settled solids	15	14	15	17	21	22	24	25	28	26
4	Added starch	19	21	23	22	26	30	35	41	45	53
5	Added casein	18	18	22	24	34	34	36	36	66	111
6	Added rosin	18	20	23	26	28	30	32	34	35	44

putrid odors. The sample to which was added 100 p.p.m. of rosin each time a freeness test of the sample was made showed 144.5% increase in drainage time. The percentage of slime formed in this sample was almost identical with that formed in the control sample. Thus, it is probable that rosin neither aids nor inhibits normal slime formation.

DISCUSSION

In the investigation it has been shown that slime forms in some recirculated white waters and that the growth is a direct result of bacterial action. Some mills use two or three different chemicals besides weekly shutdowns to control this growth. It might be more practical to inhibit slime in paperboard mills by controlling the environment of the process waters. Experiments described in this paper showed that slime growth was inhibited by proper control of the temperature and/or pH of the recirculated white water. The recirculated white-water system kept at 50°C. exhibited no slime growth with a drop of total bacterial counts from

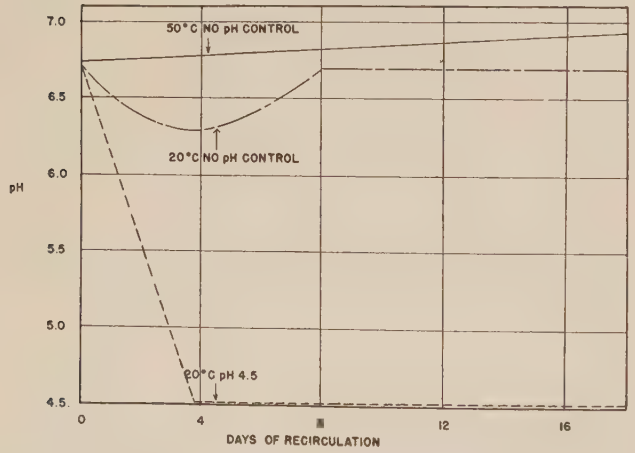

Fig. 8. pH changes of recirculated boardmill white water under controlled temperatures of 50 and 20°C.

6,200,000 to 1000 per ml. and a 35% faster initial drainage rate than white waters controlled at 10°C. The samples which were adjusted to lower temperatures exhibited some slime growth. The recirculated white-water systems controlled at a pH of 4.5 exhibited no showed that either a temperature of 50°C. or a pH of 4.5 proved an ideal environment to combat slime formation.*

The microorganisms and the food for the slime formers were found to be present in the dissolved and colloidal portion of the recirculated white water. When this portion of the white water was replaced by distilled

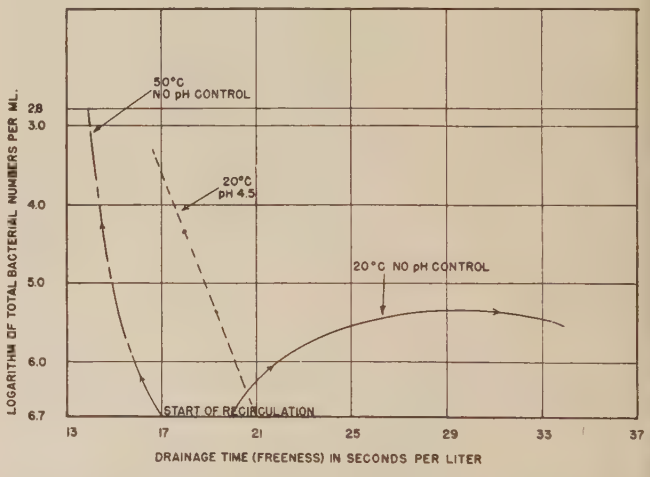

Fig. 9. Effect of controlled temperature (50 and 20°C.) or controlled pH (4.5) upon the 2-day total bacterial numbers in recirculated boardmill white water

water, no slime growth took place. Some slime microorganisms or food or both were found to be enmeshed in the settleable solids portion of the recirculated white water because the slime growth was depressed 50% by replacing this fraction of the water with fresh pulped and dried paperboard stock. The addition of soluble starch to the white water increased the slime growth 31%. Starch and other forms of available carbohydrate foods are usually plentiful in white waters and this fact is borne out by the small increase in slime growth caused by the addition of the starch. The finding that addition of casein stimulated slime growth and caused putrid odors might have been expected, but nitrogenous food matter is always low in white

* The effect of alum on recirculated white waters added at various temperatures and at different pH values will be discussed in a subsequent paper.

waters. The casein added, serving as a source of nitrogenous food for the slime forming microorganisms, resulted in an increased slime growth of 237%. It is apparent that white waters with an ample supply of nitrogenous matter can be expected to promote excessive slime growth.

The introduction of a new method of measuring the extent of slime growth may meet with some criticism. For instance, other factors besides slime growth, such as the extent of beating the fibers receive, slight changes in temperatures, operational difficulties of vacuum fluctuations, and the cleanliness of the screen, will cause a recirculated white water to drain more slowly. But if every sample is prepared alike, except for the one variable to be studied, this apparatus can be used with remarkable accuracy and consistency, the writers believe. Moreover, comparable results can be expected if an initial freeness reading is made on each sample, and the percentage decrease in freeness is recorded under controlled conditions after slime has occurred; and little doubt should exist as to the origin of the increase in drainage time.

SUMMARY AND CONCLUSIONS

Recirculation of white water is a common practice in most paperboard mills as a method of pollution abatement and of conservation of fiber, filler, and water within the mill. Slime is one of the major nuisances which result from the recirculation. Several chemicals are now being used as well as regular machine and mill clean-ups to inhibit slime growth.

A laboratory handsheet machine was designed and constructed to measure the rate of drainage of recirculated white-water mixtures.

White water obtained from paperboard mills was recirculated for various periods, subjected to pH and temperature control, sterilized or various types of food for biological growth added to determine the individual effects of treatment.

The results obtained by experimentation led to the following general and specific conclusions:

1. Slime growth in the white water decreases the drainage rate (freeness) of the white water allowing the slime growth to be measured directly by the developed laboratory handsheet machine.

2. The simple mechanical act of continuous recirculation of the white water in itself is not responsible for a change in the character of the water resulting in a decrease in freeness. It is possible that the increased dissolved solids during recirculation become susceptible to bacterial action if the white water is allowed to stand for a considerable time or remains in contact with "dead spots" in the lines during recirculation.

3. The build up of solids in laboratory recirculated white water was caused by an increase in dissolved or colloidal matter. The recirculated white water in which contamination (food and seed) was present showed a decrease in freeness.

4. Regardless of the initial pH of the white water (from 4 to 8) and despite the physical treatment of the water (intermittent stirring or quiescent conditions), the pH values tended to reach an equilibrium range between 6.2 and 7.2 with one exception, but not necessarily after the same time of recirculation or storage.

5. In the absence of pH control, slime formation took place after 5 days and the freeness of the white water decreased. This water also had a foul and fishy odor.

6. No slime growth resulted when the pH of the water was controlled with alum or sulphuric acid to 4.5.

7. Slime formation was affected by the temperature by the recirculated waste white water. Optimum temperature for slime build up was between 20 and 30°C. No slime growth occurred from 37.5 to 50°C. after 22 days. The freeness of the stock was affected by temperature from a purely physical standpoint. The higher the temperature, the faster the stock drained. About a 35% increase in freeness resulted in increasing the temperature of the recirculated white water from 10 to 50°C.

8. Characteristic pH curves were obtained. The microbiological life adjusted itself to conditions suitable for slime growth (lowering of pH to 6), after which the pH rose above 6 and slime occurred.

9. Bichloride of mercury inhibited bacterial action and also prevented a decrease in freeness of the recirculated white water. When the bacterial action was not supressed, the freeness decreased and the pH rose above 6 simultaneously showing that slime growth in paperboard mill white-water wastes can be measured by the freeness of the water.

10. A nutrient agar 2-day plate count of the total bacterial numbers in recirculated white water indicated that the numbers decreased slowly and slightly despite slime formation. However, when slime formation was inhibited by controlling the temperature at 50°C. or the pH at 4.5, these numbers decreased immediately and to a very great extent, ending at a much lower level than when slime was not inhibited.

11. The increase in time of drainage caused by slime formation varied from 20 to 250%. There was some limiting factor in boardmill white waters which, when present, caused an abundance of slime formation and when absent resulted in little or no slime. This limiting factor appeared to be present in the colloidal or dissolved portion of the white water.

12. The source of added nitrogenous food in the white water caused slime growth to proceed vigorously after a normal sliming during the first 10 days. Rosin added to the white water did not affect normal slime formation. Starch added regularly to the white water caused 31% more slime to form than would normally occur.

13. Removing the settleable solids and replacing them with fresh, dried paperboard stock depressed slime growth about 50%.

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Paper of the Journal Series, New Jersey Agricultural Experiment Station, Rutgers University, the State University of New Jersey, Department of Sanitation.

The Preparation of a Water-Resistant Starch Coating by the Use of Antimony Salts

N. M. FRISCH, M. L. CUSHING and L. GUSSMAN

A new method for insolubilizing starch-base paper-coating colors is discussed. An antimony salt, potassium pyroantimonate ($K_2H_2Sb_2O_7$) is blended with the regular components of a starch-clay coating color, applied on regular coating equipment, and dried at paper machine drying temperatures to produce a coated paper with good wet-rub properties. The theory of the chemistry of the reaction between starch and the antimony salts is discussed, and the best type of paper-coating starch for use with this process is indicated. Formulations for coating colors for high and low solids coating are given. The effect of pigments, plasticizers, drying-time, drying temperature, and pH on the antimony-starch reaction are discussed in some detail. The major advantage of the process over existing insolubilizing methods, the fact that pH adjustment is unnecessary, is brought out. The antimony-starch reaction is most effective in the 7.0 to 7.8 pH range, and that pH range coincides with maximum clay color fluidities. Since most printing processes are most effective on coated paper with coatings in that pH range, this factor aids printing and ink drying. The insolubilizing effect is evident immediately after the coating and drying process. Drying time and drying temperature are the critical factors in the efficiency of the process and several charts are presented showing the wet rub of the coating in terms of drying temperatures and time. Shelf life of the finished coating color is shown to be good and chemicals for varying the viscosity over a wide range are discussed. The use of potassium pyro antimonate as insolubilizing agent raises the Dennison wax test over the wax of the same color, without the antimony salt. Several forms of the antimony salt are available: dry, white powders, and stable, water-thin, straw-colored 40% solids solutions. Other uses of the starch-antimony reaction are indicated including application for insulation and wallboard coating, particularly where fire-retardent coatings are desired. Other fields where experimental work is in progress are wet-strength papers, asbestos paper, and wallpaper.

THE use of starch as an adhesive in machine coating achieved its first wide acceptance soon after the beginning of this century. To produce a coating in which starch is used and which is sufficiently water resistant for lithographic printing requires a special modification of the starch with some agent which will serve to insolubilize the starch adhesive.

Among the various modifications of starch adhesive there may be listed resins such as urea-formaldehyde (*1*), melamine formaldehyde (*2*), treatment with chromium salts as described by Caesar (*3*) and Iler (*4*), or by the use of latices such as Dow 512K as described by Stilbert (*5*). A recent monograph (*6*) contains a section which summarizes the patents in regard to water-resistant starches.

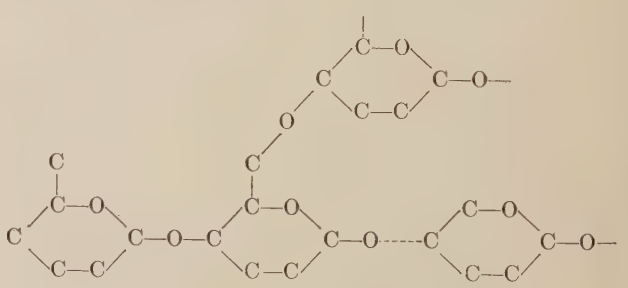
The object of this paper is to more fully expand on the application of the reaction of potassium pyroantimonate as a starch insolubilizing agent as presented by Griffin, Truax, and Nuttall (*7*) to the problem of producing water-resistant starch coatings.

The original and commercial developments of this reaction were in the textile field, but the chemistry or mechanism of the reaction is of interest for application in many other fields which require the use of insolubilized starch.

In considering what may be the mechanism of the reaction of insolubilization of starch by antimony salts it is necessary first to consider the nature of the starches used.

Unmodified corn starch is composed principally of 25% of amylose or A fraction and 75% of amylopectin or B fraction (*8*). Other starches such as wheat, tapioca, and potato do not differ materially from this composition (*9*). Waxy maize starch on the other hand is more nearly completely composed of amylopectin (*10*). It has been shown that the amylose fraction can exist in at least three crystalline orientations as depicted by x-ray diffraction data. The A and B x-ray diffraction pattern (*11*) is thought to be caused by "fiber bundle" type of orientation, while the V pattern is caused by a helix form of structure (*11*). Amylopectin appears to be so highly branched as to prevent ready orientation into any fixed pattern, thus giving rise to an amorphous character (*11*). Since this form predominates in whole raw starches it is probable that any explanation of the reaction mechanism should be based on the structure of amylopectin rather than on that of amylose.

It is fairly well accepted that starch (and cellulose) binds water, alcohols, and other starch molecules to itself by means of secondary valences known as "hydrogen bonds" (*12*). Each of the oxygen atoms in the many glucose residues composing starch is an "acceptor" for hydrogen bonding and a potential seat of binding action. A picture of the amylopectin molecule must show the multiple-branching apparently present and will look like this



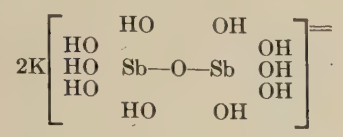
which may be written as $(C_6H_{10}O_5)$ or five sites of hydrogen bonds. Potassium pyroantimonate has ap-

N. M. FRISCH, M. L. CUSHING, and L. GUSSMAN, Stein, Hall & Co., New York, N. Y.

proximately the composition which would arise from the following reaction

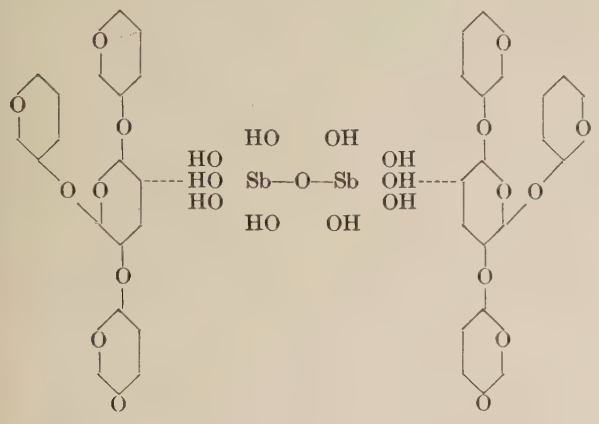
$$2KSb(OH)_6 \rightarrow K_2Sb_2O(OH)_{10} + H_2O$$

Each of the 10 hydroxyl groups on the pyroantimonate molecule has a potential hydrogen bond "donor" in its H atom. The molecule may be written as follows, with valence angles omitted in order to simplify



In such a molecule the only groups available for reaction are the hydroxyl groups (OH) which may form hydrogen bonds with such molecules as starch or which conceivably could by sufficient energy be driven to form an O—Sb—O—C linkage by loss of water between glycosidic OH and pyroantimonate molecules.

Because of its two antimony atoms the pyroantimonate molecule can be considered difunctional and can serve as a cross-linking or bridging element between two individual starch molecules. Represented schematically it may result in the following spacial grouping.



If it were necessary to utilize one pyroantimonate molecule per two starch molecules the weight ratio would be 1.5:1. Actually 3 to 5% is adequate to render the starch substantially water resistant. This happens to be in the range of the proportion of one pyroantimonate per branch of amylopectin which might indicate the necessity for insolubilizing or immobilizing each one of the branches in amylopectin and also to a lesser extent forming "cross links" among amylose molecules.

Laboratory results and field trials have indicated that it is necessary to utilize approximately 10% potassium pyroantimonate on the weight of starch to produce a paper coating having satisfactory water resistance.

The amount of potassium pyroantimonate has to be balanced between cost of treatment, gelling tendencies with higher amounts of chemical, and increasing water resistance with increasing amounts.

The effect of the nature of the starch on the degree of water resistance can be stated in the general terms that the less degraded the starch the greater the water resistance.

The longer and more complex molecule of native or untreated starch has more groups which can combine with the cellulose surface and with the pyroantimonate. Once this complex is laid down it will be less soluble or more water resistant due to the larger number of hydrogen bonds formed.

However, native starches form water dispersions which are high in viscosity and take too much water to permit their use as coating starches. Thus some type of-modification of structure is necessary.

Various means may be used to bring about the desired modifications such as wet acid conversion, dextrinization, oxidation (chlorination), and enzyme conversion. Differences within each of these classes are most clearly related to solubility. The more readily soluble products do not give good water resistance with pyroantimonate. It is believed that this relates to the decrease in chain length with its concomitant decrease in hydrogen-bonding effect. A point is finally reached where the molecular size of the dextrin is so small and solubility so easy that no amount of pyroantimonate will serve to render the complex insoluble.

Among the various starch-converted products the lower the solubility the better the water resistance. Thin boiling, white dextrines, and oxidized (chlorinated) starches differ in the degree to which they may be insolubilized by this process. In general the chlorinated starches show slightly poorer results which may be due solely to chemical structure differences of these products.

In the work in the textile field it has been observed that there is a slight but significant increase in the degree of water resistance when comparing the effect of potassium pyroantimonate on corn starch and amioca. This would indicate that an increase in the degree of branching of the carbohydrate molecule leads to greater reactivity with the insolubilizing agent—or at least an increase in the energy required to redissolve the complex.

The following experimentally determined facts may be set forth:

1. A higher degree of insolubilization results if the potassium pyroantimonate is present during the entire cooking operation rather than when it is added at the end of cooking schedule which would infer that some initial stages of the reaction take place during the "opening" of the starch. However, if the pyroantimonate be present during the entire cooking schedule one must anticipate possible difficulties due to increase in viscosity and a tendency to gel. This may be offset by the use of the proper starch, or by the addition of plasticizers such as urea, or dicyandiamide.

2. Whatever may be the mechanism of this initial reaction the complex formed does not interfere with enzymatic conversion of the starch. (This is at 10% levels of pyroantimonate—no information is available as to the effect of higher concentrations.)

APPLICATION OF THE ANTIMONY STARCH REACTION TO PAPER COATING

Since there are several basic differences between the formulations used in the textile and paper industries, it became necessary to re-evaluate the chemical reaction in the presence of the chemical systems com-

monly used in the paper industry. The two major differences in "color" compounding in the two industries are (1) the fluidity of the starch used, and (2) the adhesive to pigment ratio. Preliminary tests showed that substantially larger quantities of potassium pyroantimonate, based on the starch, are required to achieve good water resistance, and it was shown that this increase is, in large measure, due to the fact that the paper-coating industry utilizes comparatively highly converted starches.

Choice of Adhesive

Three major factors govern the choice of adhesive for a coating color, (1) the type of coating equipment, (2) the per cent total solids of the color formula, (3) the adhesive strength requirement of the coated sheet.

Having determined the proper fluidity range for the color, there are several types of starch conversions from which to choose.

Depending on such factors as the cooking and handling equipment, the skill of the labor available, and economic considerations, the following starch products are available, each being supplied in several fluidities: (1) oxidized (chlorinated) starches, (2) dextrines (heat and/or acid conversions), and (3) enzyme conversions of raw starches (corn, potato, tapioca). Typical samples of each group noted were tested for reactivity with potassium pyroantimonate. The data in Table I show that except for the chlorinated starches products generally accepted in the industry are satisfactory for use in this process. The reactivity of a starch with potassium pyroantimonate decreases with increasing degree of conversion. For example a white dextrin in the 90 fluidity range is less reactive (in terms of water resistance of the coating) than a starch which has been converted by the same process only to 30 fluidity.

Water Resistance

The water resistance of the coating is determined by the following method.

Table I. Coatings Using 10% Modified Antimony Salt

Dried at 220°F. for 2 minutes, Casein coated paper water resistance rated as 100%

Starch	*Starch-clay, %*	*Enzyme, %*	*Total solids, %*	*pH*	*Brookfield viscosity 30 r.p.m., 25°C.*	*Wax*	*Water resistance, %*
RH6P	20	...	50	7.46	1096	7	88
Supercote No. 4	15	...	49	7.38	785	6	91.5
Supercote No. 10	25	...	51	7.50	890	8	90
Superfilm No. 6	20	...	50	7.50	788	7	81
Enzo 7	15	0.35[a]	50	7.59	696	Edge 4–5 full 6	88
Enzo 7	15	0.85[b]	50	7.24	892	Edge 5 full 6	93
Idaho potato	15	0.70[b]	50	7.39	500	Edge 6 full 7	91
Idaho potato	15	0.50[b]	50	7.65	754	7	95
Amioca	15	0.50[b]	50	7.47	557	7	91.5
Amioca	15	0.35[b]	50	7.42	1480	7	93

[a] Amyliq concentration.
[b] Vanzyme No. 1, dry.

A 2 by 2 inch square of the coated paper is conditioned at 72°F., 50% R.H. and weighed. One drop of distilled water is placed on the coated surface of the paper and the wet spot is rubbed 10 times with uniform pressure. The rubbed portion is then rinsed with distilled water to remove the loosened particles. The square is then reconditioned and reweighed. The loss in weight is fitted into a scale which is determined by rating the water resistance of casein-coated paper as 100% and that of a starch-coated paper without antimony salts as 0%.

As noted above, the chemical conversion process produces a product which is less easily insolubilized by antimony salt. However, water-resistant coatings equivalent to those produced with less degraded starches can be obtained when using highly converted starches by increasing the starch to pigment ratio. This not only produces improved water resistance but also better dry adhesive strength as measured by the Dennison wax test.

Table II. Effect of Varying Amounts of Potassium Pyroantimonate in a Coating Color

Coatings made with RH6P

Starch clay, %	*Sb salt, %*	*Solids, %*	*pH*	*Brookfield viscosity 30 r.p.m., 25°C.*	*Thixotropic index 6 r.p.m./60 r.p.m.*	*Wax*	*Water resistance, %*
20	0	50	7.45	325	2.33	Edge 5+, full 7	0
20	2	50	7.48	350	2.66	Edge 6, full 7	47
20	4	50	7.48	440	3.25	Edge 6, full 7	79
20	6	50	7.40	685	4.17	Edge 6, full 7	84
20	8	50	7.32	1140	4.69	7	87
20	10	50	7.46	1096	4.65	7	88
20	15	50	7.57	1640	5.02	Edge 7, full 8	88

Choice of Pigments

As clay is the most widely used pigment in the coating industry, most of the experiments were made on clay colors. However, colors incorporating TiO_2 and $BaSO_4$ were checked, and it was found that they water-

proofed equally as well as clay colors. However, it was observed that, as the percentage of $CaCO_3$ in the coating color was increased, the water resistance was decreased. This effect can be counteracted by increasing the potassium pyroantimonate starch ratio or by increasing the adhesive-pigment ratio.

The Potassium Pyroantimonate Ratio

For coating color formulations, it was found that 10 pounds of potassium pyroantimonate per 100 pounds of adhesive is optimum to produce satisfactory wet rub. Table II and Fig. 1 show the effect of varying the

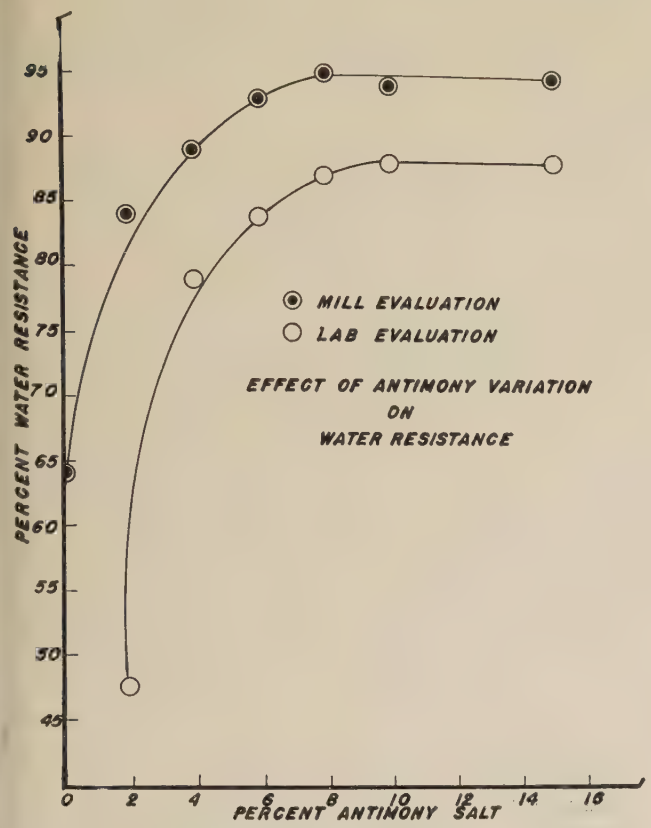


Fig. 1. Independent mill and laboratory evaluations of the effectiveness of antimony salts on water resistance

Coatings were dried at 220°F. for 2 minutes. Mill rates untreated starch coating as 64% water resistant, whereas laboratory rates this as 0%. Color pH = 7.32 — 7.57.

amount of potassium pyroantimonate in a coating color. It is evident that with increasing amounts of potassium pyroantimonate not only the wet rub, but the wax test increases. Along with these improvements, viscosity and thixotropy increase. The establishment of water-resistance standards therefor, for the particular coated sheet under test, will determine the potassium pyroantimonate-starch ratio. Since viscosity and thixotropic properties of a color are often critical, a study was made of methods to counteract viscosity changes introduced by the use of potassium pyroantimonate. Several commonly used chemicals, including urea and dicyandiamide are effective in reducing the viscosity and thixotropy of potassium pyroantimonate containing colors. Results are shown in Table III. Another method for controlling the end viscosity of a color is by the choice of adhesive of the proper degree of conversion.

Table III. Dicyandiamide as fluidifier for antimony starch coating.

Dicyandiamide, %	*Brookfield viscosity, cp.*				
	Immed.	*1 hr.*	*2 hr.*	*3 hr.*	*20 hr.*
0	2950	4800	4900	4750	4450
1	2700	3600	3700	4050	5600
2	2750	3600	3950	3750	5800
3	2600	3300	3800	3700	3400
5	2550	3050	3800	3650	3050

Coating composition
480 g. Lustra clay, 256 cc. H_2O, 24 ml. 10% TSPP
86.4 g. Supercote No. 10, 149.6 ml. H_2O
6.91 g. Antimony salt, (80% active principle)
Starch-clay ratio = 18%
Total solids = 57.5%

Method for Preparing Potassium Pyroantimonate Coating Colors

Few changes in the normal procedure are required to prepare a potassium pyroantimonate starch color: (1) The clay is slipped in the usual way (in some cases it has been found advisable to substitute the corresponding potassium salts for the common dispersants such as TSPP, Calgon, etc. (2) The starch is cooked or converted in routine fashion. (3) The pigment slip and the adhesive are blended. (4) At this point, immediately after the pigment and adhesive have been blended, the required amount of potassium pyroanti-

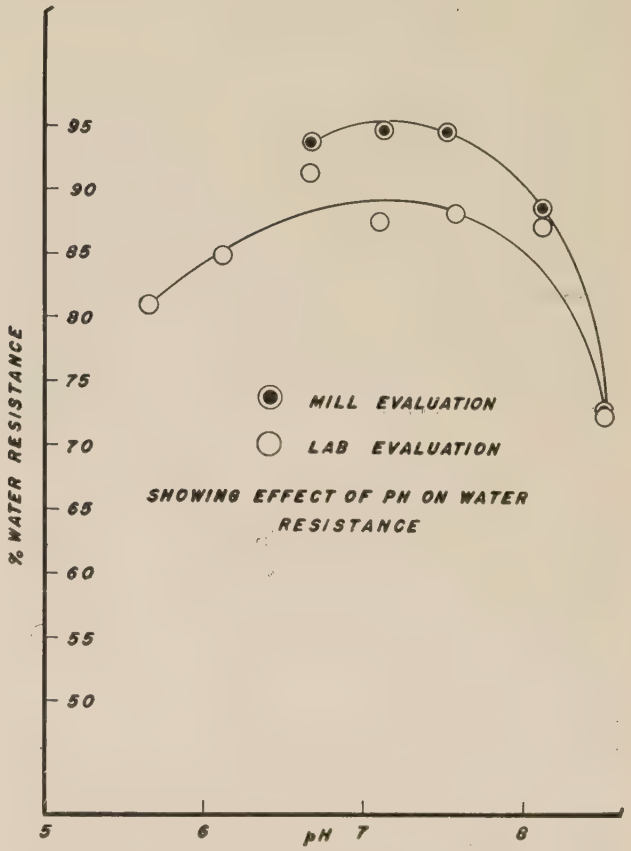


Fig. 2. Coatings made with 10% of modified antimony salt on starch, dried at 220°F. for 3 minutes

monate is added. Then color, defoamer and/or plasticizer, if needed, are added. (5) Finally, the color pH is adjusted to fall within the 7.0 to 7.7 range. Figure 2 shows this to be the preferred range for best water resistance. Thus, except for the last two steps, no changes are required in normal mill practice.

Time-Temperature Requirements

Temperature is the key to the potassium pyroantimonate reaction. While a measurable degree of water resistance can be developed by air drying, water-resistant coatings with commercial possibilities require drying temperatures on the order of 220°F. for from 30 to 120 seconds. Figures 3 and 4 illustrate the effect of time and temperature on the potassium pyroantimonate-starch reaction that produces starch insolubilization. The temperatures required to complete this reaction are available on the average paper machine. However, some coaters are so located in the drier section that the drying time is too short. However, any combination of drum drying, calendering, or roll storage that will maintain sheet temperature of 200 to 220°F. for the required time will produce satisfactory insolubilization. For example, a coated sheet with good water resistance has been produced on conversion coating equipment equipped with modern-type tunnel drying. The coated roll was promptly supercalendered. The combination of tunnel drying, supering, and roll storage was sufficient to complete the reaction. Several methods of increasing the drying temperature and the drying time of the coated sheet are available ranging from increasing steam pressure in the drum drier, to the installation of additional driers or the use of infrared lamps set up between the existing driers.

Interfering Chemicals

Some chemicals and pigments interfere with the starch potassium pyroantimonate reaction. During evaluation tests of plasticizers, it was found that stearate soaps cannot be satisfactorily used in potassium pyroantimonate colors since they inhibit the insolubilization reaction. A new plasticizer called Cetosol K was developed for use in this color. The preservatives Merfenol (*13*) and Lignasan (*14*) were tested for use in potassium pyroantimonate color. Both are compatible, but the latter reduced the water resistance somewhat. It was found that the antimony insolubilization of starch may be utilized with latices and also flameproofing formulations without detriment to either process.

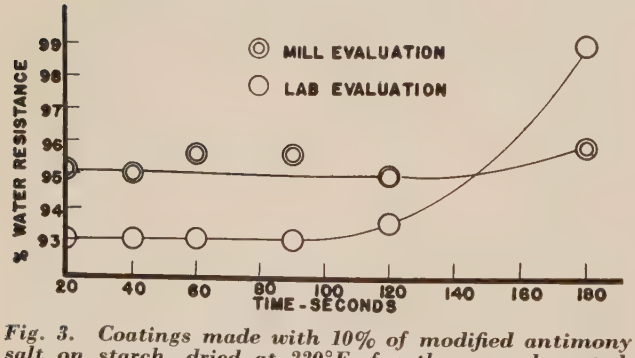
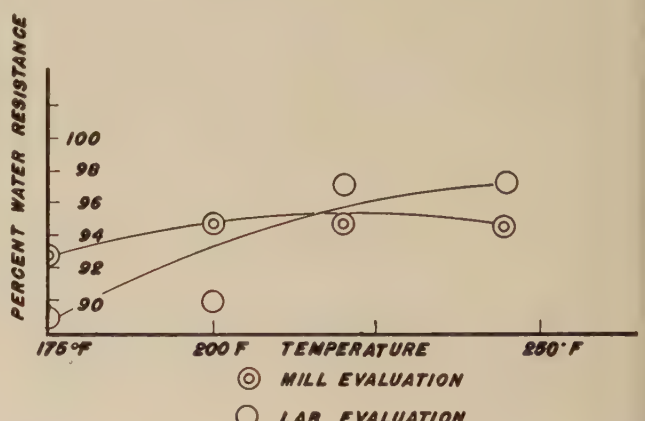


Fig. 3. Coatings made with 10% of modified antimony salt on starch, dried at 220°F. for the seconds noted. Color pH = 7.46

Service Life of Potassium Pyroantimonate-Starch Colors

At normal storage temperatures in tanks equipped with agitators potassium pyroantimonate colors will generally be as stable as the comparable starch colors. In high solids colors where a viscosity build-up results from potassium pyroantimonate addition, the use of small amounts of urea or dicyandiamide (1 to 3%) eliminates that problem.



EFFECT OF TEMPERATURE ON WATER RESISTANCE.

Fig. 4. Coatings made with 10% of modified antimony salt on starch, dried for 2 minutes at the indicated temperatures. Color pH = 7.46

In low solids coating, where the color is prepared in batches to run for 4 to 5 hours, and the batch is maintained at 170 to 180°F. for that time, no viscosity difficulties were experienced.

Utilization of the Starch-Antimony Reaction in the Paper Industry

This process is now in the stage of commercial development. Field trials in various phases of the coating industry give promise of reduction to commercial production.

Examination of the water resistance obtainable with pyroantimonate-starch coatings and equipment available in the paper industry indicate the following possible fields of application: coated paper and paperboard, coated wallboard, wallpaper, asbestos paper, and wet-strength paper.

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Steam-Jet Vacuum Refrigeration for Control of Sulphite Acid Composition

E. F. DHUS

The temperature of the water used in making sulphite cooking acid of the proper composition is important. Refrigeration is sometimes required to obtain the correct temperature. The advantages and principles of operation of steam-jet vacuum refrigeration are given. The installation and operation of the system at the Hammermill Paper Co. is described.

THE acid used for manufacturing sulphite pulp is composed of a bisulphite and sulphurous acid. Uniform, efficient operation of the pulp mill, and the quality of the pulp depend on a proper acid composition.

Temperature is an important variable in determining the composition of the acid which is made by passing water over limestone in the presence of sulphur dioxide gas. When the sulphur dioxide is absorbed by water, sulphurous acid is formed, and some of it reacts chemically with the limestone to form calcium bisulphite. The rate of this chemical reaction varies directly with the water temperature; the higher the temperature, the larger the percentage of calcium bisulphite in the acid.

The percentage of sulphurous acid also depends on the temperature of the final acid; a greater amount of sulphur dioxide will be absorbed at a lower temperature and thus produce an acid with a larger percentage of sulphurous acid. An exception to this is when the acid is produced and stored under pressure.

Recent practice in the sulphite pulp industry has been to increase the percentage of sulphurous acid. The benefits from doing this have been well demonstrated. It was not always possible to obtain the desired acid composition in the summer with existing equipment.

The temperature of the water for making acid of the desired composition is between 60 and 65°F. Water for acidmaking varies in temperature at the point of use from 35°F. in winter to 85°F. in summer. It is necessary to cool the water for about four months each summer a maximum of 20°F. Three hundred gallons per minute are required 24 hours a day, 6 or 7 days per week.

STEAM-JET VACUUM REFRIGERATION

Various types of refrigeration were investigated. Steam-jet vacuum refrigeration has the following advantages (*1*): (1) lower initial cost than other types, (2) lower operating cost, (3) lower maintenance cost because of no moving parts, (4) no replacement of refrigerant, (5) no pressure in system, (6) no housing required, (7) no additional operator is required since the acidmaker can operate the plant along with his other duties, (8) low-pressure steam can be used. Low-pressure or exhaust steam is used in the mill for heating. Use of low-pressure steam in the refrigerating unit, which is to be used only in summer, will keep the low-pressure steam load in better balance through the year.

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Principle of Operation

The principle of operation of steam-jet vacuum refrigeration is based on the fact that the boiling point of a liquid depends on the pressure to which it is subjected; lower pressure lowers the boiling point. A vacuum reduces the boiling point from that at atmospheric pressure. If a liquid with a temperature corresponding to its boiling point is sprayed into a closed vessel being held at a lower pressure, the entering liquid is above the boiling point at the new pressure, and evaporation takes place.

During the process of evaporation, the liquid must absorb heat equivalent to the latent heat. This is very large compared to the sensible heat. For example, it requires only 10 B.t.u. to cool 1 pound of water from 40 to 30°F., while to evaporate 1 pound of water at 40°F. requires 1069 B.t.u. If the heat is not available from some external source it will take it from the remaining liquid. The conversion of latent heat to sensible heat cools the remaining liquid, the process continuing until the liquid is at the temperature corresponding to the boiling point at the vacuum maintained. The amount of liquid required for evaporation is very small; only 1% of the total is required to cool water from 50 to 40°F.

Table I shows the pressure-temperature relation for steam at temperatures useful for cooling.

In the evaporator, or flash chamber, where the above process takes place, a high vacuum is maintained by the action of a series of steam-jet ejectors and condensers. Steam flowing at high velocity through nozzles in the steam-jet ejector head is expanded in a venturi-shaped diffuser. The kinetic energy of the high-velocity steam is partly utilized in imparting velocity to the water vapor evaporated from the water being cooled, and partly in compressing this vapor from the evaporator pressure to the condenser pressure.

Condensation of the steam used in the ejector, plus the vapor evaporated to perform the cooling, can be performed in either a surface or a barometric condenser. The surface condenser is used where it is necessary to recover the condensate without contamination. However, a barometric condenser is much cheaper to install and maintain, and requires less floor space but more head room; the bottom of it should be at least 34 feet above ground level.

The initial evacuation of the evaporator, or flash chamber, and the constant purging of air and other noncondensible gases are performed by a secondary

Table I. Pressure-Temperature Relations for Steam

Water Temperature °F.	*Absolute pressure, in. mercury*	*Vacuum referred to 29.92 inches mercury barometer*
40	0.248	29.673
50	0.362	29.559
60	0.522	29.399
70	0.739	29.183
80	1.033	28.888

group of steam-jet ejectors and condensers. These are relatively small in size. They consist of two steam jets in series with an intercondenser. These, in turn, are in series with the primary jet and condenser on the flash chamber.

Description of Equipment Installed

A Worthington steam jet-type refrigeration unit designed to cool 300 g.p.m. of water from 85 to 65°F. was installed in 1948. Its rated capacity is 250 tons of refrigeration.

Cooling takes place in a flash chamber which is a horizontal cylinder 54 inches inside diameter by 13 feet long, divided into two compartments, and located 26 feet above the ground. Two large booster ejectors, each rated at 125 tons of refrigeration, are connected directly to the flash chamber, one to each compartment. Two boosters and two compartments in the flash chamber are used to make it possible to handle part loads more economically. The diffusers from these point upward and connect into the side of the barometric condenser which is a vertical cylinder 48 inches inside diameter by 14 feet 6 inches long, with a 34-foot dropleg down to an 11-foot square hotwell under the flash chamber. A size 3 CPD single element, two stage, secondary steam-jet air ejector with a barometric type intercondenser connects to the top of the large primary barometric condenser.

The temperature of the water leaving the flash chamber is regulated by automatically opening and closing the steam valve to one of the booster ejectors. The steam must be either on or off since the boosters will not operate with a partial steam supply.

Two pumps are required; one to pump the cooled water to the top of the acid towers, and the other to transfer the mixture of condensate and condenser water to the point where it is reused.

The unit requires 4000 pounds of steam per hour at 25 pounds gage pressure when both of the primary booster ejectors are in operation, and 220 pounds of steam per hour at 125 pounds gage pressure for the secondary air ejectors. The ejectors are designed for steam at these pressures and will not function below that point. It is necessary to set the design pressure at the minimum pressure that will exist at the point of installation. The steam line pressure for the primary booster ejectors is usually 30 to 32 pounds gage but may occasionally fall to as low as 27 pounds gage. Since the steam consumption is proportional to the absolute pressure, a pressure reducing station was installed at the unit to reduce the pressure to 25 pounds gage with any line pressure above 27 pounds gage. In this way, the unit is certain of having the necessary steam pressure for operation without using excessive steam when the line pressure is higher than design pressure. The steam consumption of the secondary ejectors is so low that a pressure reducing station was not installed for them.

Water required for condensing amounts to 1540 g.p.m. for both condensers at maximum load and at the peak temperature of 85°F. With lower water temperature and part load, less water is used.

Steam-jet refrigeration requires more cooling water than compressor-type refrigeration using a chemical refrigerant. However, by keeping the water system enclosed, the water is kept clean and can be used again. The temperature of the water leaving the condenser is regulated to 95°F. This warm water, together with the warm water from the relief and burner gas coolers, is pumped back into the mill water system at points where warm water is desirable for washing stock in the blowpit and in the bleach plant.

Operation of the Refrigerator

Operation of the unit is very simple, and since it has automatic controls, practically no attention to it is required once it has been started. The acidmaker operates it along with his other duties.

About 20 minutes are required to start the unit.

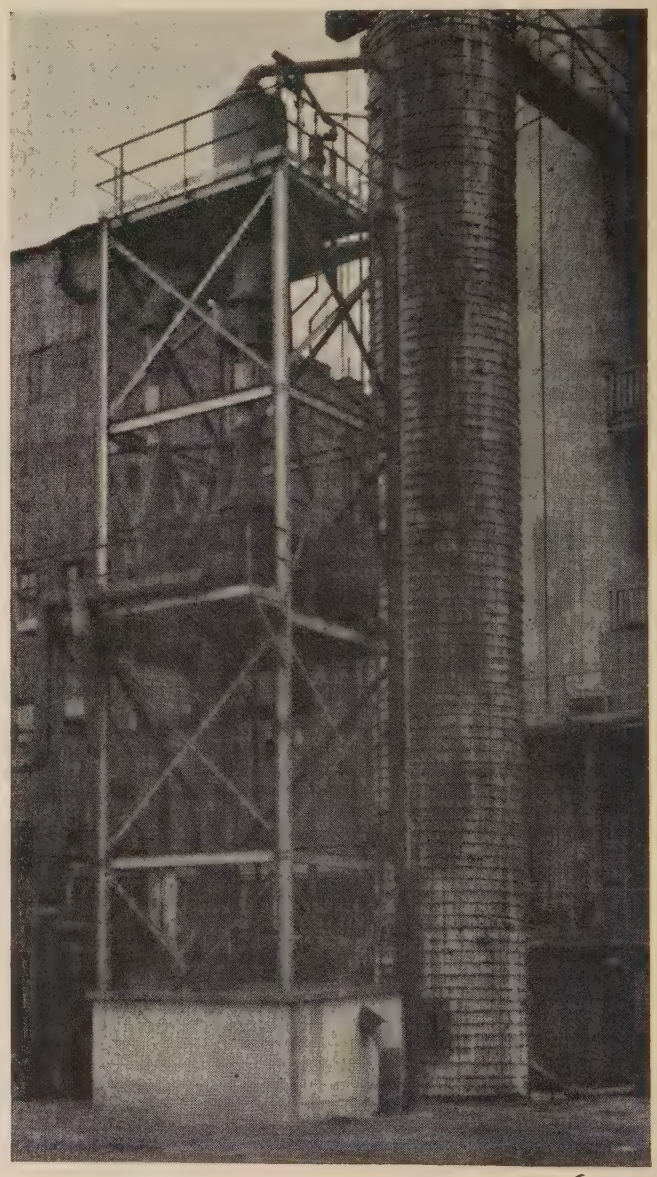

Fig. 1, Steam-jet vacuum refrigerator

This is the time required to evacuate the flash chamber and barometric condenser of air before full operation is started. The high-pressure steam to the secondary air ejectors, the condenser water to both condensers, the water to be cooled, and the pumps are all started. When the absolute pressure is reduced to 1.8 inches mercury, the steam to the primary booster ejectors is turned on, the automatic control set, and the unit is in full operation. The only attention required while the unit is operating is to adjust the condenser water supply occasionally to compensate for incoming water temperature changes. Since these changes are neither rapid nor often, this does not require much attention. The condensing water to the primary condenser is adjusted to 93 to 95°F. outlet temperature, and the water to the secondary condenser to 105 to 110°F. outlet temperature.

RESULTS

The composition of the acid was maintained according to specification all through the summer of 1948, the first year after the unit was installed. The addition of the steam-jet refrigerator has made it possible to increase the strength of the digester acid with a minimum capital expenditure and operating cost without adding to the burden of the operating crew. The increased acid strength has brought about a more uniform and efficient operation of the pulp mill and improved the quality of the pulp produced.

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The Groundwood Pulping of Mixtures of White Spruce and Quaking Aspen

E. R. SCHAFER and A. HYTTINEN

Groundwood pulping experiments were made on quaking aspen and white spruce and on three mixtures of these woods. As the amount of aspen in the mixture was increased, the motor load remained constant but the grinding rate increased, energy consumption decreased, freeness increased, and strength decreased. These relationships were linear, or nearly so, in most instances. The properties of mixtures of spruce and aspen groundwood pulp were, in general, the same whether the woods were ground together or ground separately under the same conditions of grinding.

Although spruce and other softwoods are still the main woods used for groundwood pulping, local supplies are diminishing and shipments being received by mills from greater distances are more costly. The use of aspen for groundwood pulping is increasing, and both spruce and aspen are being used in many mills. Since the grinding characteristics of the two species are different, it is preferable to grind them separately. It is not always feasible, however, to handle and to grind them separately. In such cases they are ground together in mixtures of varying proportions. This report presents the results of a limited number of experiments made at the Forest Products Laboratory on the grinding of mixtures of spruce and quaking aspen.

EXPERIMENTAL PART

Wood Used

The spruce wood available for these tests averaged 4.8 inches in diameter, 46 years in age, and 26.7 pounds per cubic foot in density (moisture-free weight and green volume). Since this wood was seasoned and quite dry when received, it was wetted to a moisture content of about 50% before being used. The moisture content of the aspen was about 45%. The aspen averaged 5.4 inches in diameter, 25 years in age, and 23.4 pounds per cubic foot in density.

The moisture content of wood is a factor affecting grinding characteristics and pulp properties and should be considered in grinding mixtures as well as in grinding single species. In these experiments the moisture content was kept constant, and that of the aspen was approximately that of the spruce.

Grinding Equipment and Operation

The Forest Products Laboratory experimental grinder is equipped with a 54-inch diameter by 8-inch wide pulpstone. In this instance, the pulpstone was composed of a fine grit, aluminum oxide abrasive, and its structure was of the "induced-pore" type. The stone had been dressed with a 10-cut, $1^1/_2$-inch lead spiral burr followed by a 14-point diamond burr. Prior to the start of these experiments, the stone had been in use 97 hours since the last burring. The grinding surface was judged to be in a medium-sharp condition. The grinder has three pockets which were all operated simultaneously, except in runs made at the highest grinding pressure when only two pockets were used.

The groundwood pulps were screened through a flat plate screen having slots 0.008-inch wide and dewatered on a suction filter. Pulp-test sheets of about 115-pound basis weight (25 × 40—500) were made and tested by TAPPI Standard Methods. Screen analyses were made with an Appleton selective screen.

Grinding Experiments

Experiments were made on spruce-aspen mixtures containing 0, 25, 50, 75, and 100% of aspen by weight based on moisture-free wood. A series of three grinder runs, in which only the grinding pressure was varied, was made on each mixture. Grinding pressures of 20, 30, and 40 p.s.i. were used for the 50, 75, and 100%

E. R. Schafer, Chemical Engineer, and A. Hyttinen, Chemical Engineer, Forest Products Laboratory, maintained at Madison, Wis., in cooperation with the University of Wisconsin, Forest Service, U. S. Department of Agriculture.

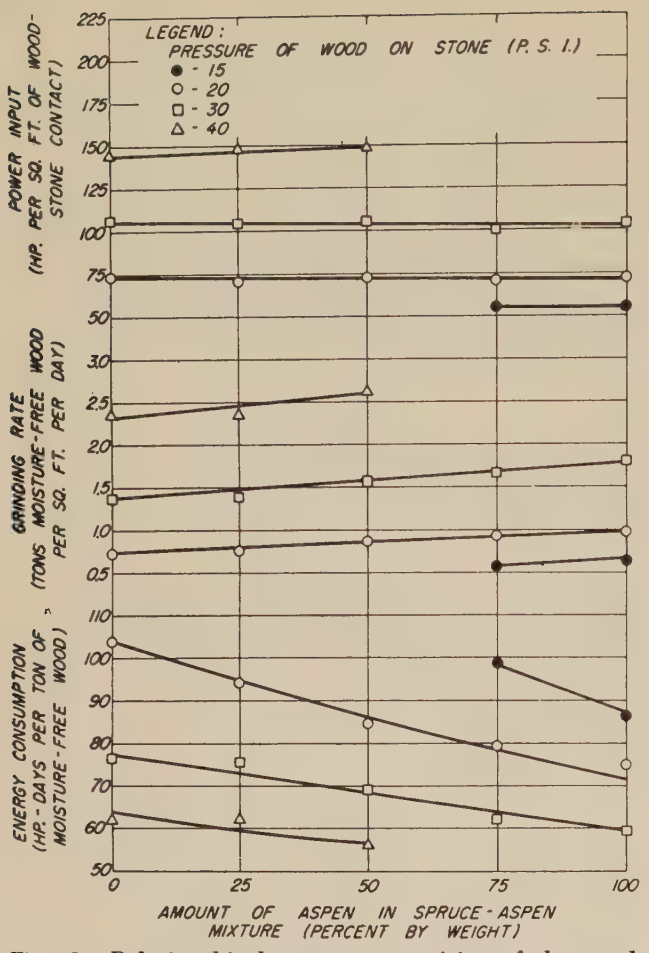
LEGEND:
PRESSURE OF WOOD ON STONE (P. S. I.)
● - 15
○ - 20
□ - 30
△ - 40
POWER INPUT (HP. PER SQ. FT. OF WOOD-STONE CONTACT)
GRINDING RATE (TONS MOISTURE-FREE WOOD PER SQ. FT. PER DAY)
ENERGY CONSUMPTION (HP.-DAYS PER TON OF MOISTURE-FREE WOOD)
AMOUNT OF ASPEN IN SPRUCE-ASPEN MIXTURE (PERCENT BY WEIGHT)

Fig. 1. Relationship between composition of the wood mixture used and certain grinding characteristics in the groundwood pulping of spruce-aspen mixtures.

spruce series; and pressures of 15, 20, and 30 p.s.i. for the remaining two series.

DISCUSSION OF RESULTS

It is well known that spruce and aspen differ in grinding characteristics and in their groundwood pulp properties. A comparison in Table I of runs 772 and 773 on spruce with runs 775 and 776 on aspen, respectively, gives a quantitative idea of these differences when the two are ground under the same conditions. It may be noted that the motor load, or power input, is the same for the two species when ground at the same pressure but, because aspen grinds faster, the energy consumption per ton is proportionately lower for the aspen. The aspen and spruce pulps also differ considerably in freeness, screen analysis, and strength.

When spruce and aspen are to be ground separately, the stone surface, and other conditions as well, should be selected so as to give the best pulp from the particular species. It is apparent that spruce can be ground satisfactorily on a sharper stone surface than aspen can. The condition of the stone surface used in this work was intermediate in sharpness. A grinding pressure of 30 p.s.i. inch with three pockets in simultaneous operation, which gave a full load on the grinder motor, produced spruce pulp lower in freeness (grinder run No. 773) and aspen pulp higher in freeness (grinder run No. 776) than is ordinary for groundwood pulps used in printing-paper furnishes.

The results of grinding mixtures of spruce and aspen are shown by curves in Figs. 1 and 2. As shown in Fig. 1, the power input is fairly constant at a given grinding pressure and is not dependent on the proportions of aspen and spruce in the mixture. The grinding rate increases and the energy consumption decreases with the aspen content of the mixture in linear, or approximately linear, relationships. Therefore, for a given set of conditions, the grinding rate and energy consumption for any mixture of spruce and aspen can be calculated from those of the individual species.

Figure 2 shows the relationship between certain pulp properties and the composition of the wood mixture used. The freeness increased with the amount of aspen in the mixture, but tensile, tearing, and bursting strengths decreased. These relationships are nearly linear, except in some instances in which the tensile or bursting strength for the pulps from the mixtures falls below a straight line joining 100% spruce and 100% aspen values. Thus it is indicated that a fair estimate could be made, from the properties of the spruce and aspen pulps ground separately, of the properties of pulp that might be prepared by grinding a spruce-aspen mixture under the same conditions.

The action in a pocket where an aspen block and a spruce block of equal size are being ground side by side at the same rate can only be surmised. The aspen block will be pulped at a slower rate than if ground with another aspen block of equal size under the same unit pressure. The spruce, on the other hand, will prob-

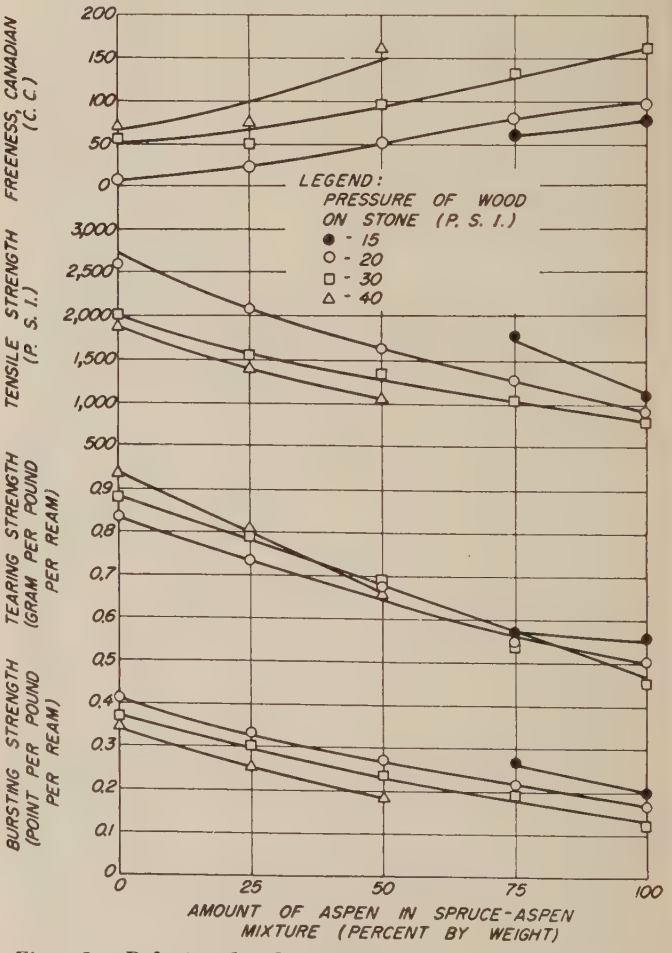
LEGEND:
PRESSURE OF WOOD ON STONE (P. S. I.)
● - 15
○ - 20
□ - 30
△ - 40
FREENESS, CANADIAN (C. C.)
TENSILE STRENGTH (P. S. I.)
TEARING STRENGTH (GRAM PER POUND PER REAM)
BURSTING STRENGTH (POINT PER POUND PER REAM)
AMOUNT OF ASPEN IN SPRUCE-ASPEN MIXTURE (PERCENT BY WEIGHT)

Fig. 2. Relationship between composition of the wood mixture used and certain pulp properties in the groundwood pulping of spruce-aspen mixtures.

Table I. The Groundwood Pulping of Mixtures of White Spruce and Quaking Aspen

Grinder run No.	Wood by weight[a] Spruce, %	Aspen, %	Grinding data[b] Pressure of wood on stone,[d] p.s.i.	Grinding rate, moisture-free wood per 24 hours,[e] tons	Power input,[e] hp.	Energy consumed per ton moisture-free wood, hp.-days	Properties of pulp suspension: Freeness Schopper-Riegler, ml.	Canadian standard, ml.	Screen analysis: Retained on 24-mesh, %	Passing 150-mesh, %	Fiber length index,[f]	Properties of pulp test sheets[c]: Bursting strength (25 × 40—500), pts./lb. per ream	Tearing strength (25 × 40—500), gm. per lb. per ream	Tensile strength p.s.i.	Density, g./cc.	Brightness, %
772	100	0	20	0.70	74	104	123	10	13.1	52.1	0.083	0.41	0.83	2560	0.45	59.4
773	100	0	30	1.39	106	76	243	55	21.4	43.3	0.099	0.37	0.88	2000	0.43	58.0
774	100	0	40	2.36	146	62	252	68	20.5	46.3	0.093	0.35	0.93	1926	0.43	60.1
780	74	26	20	0.75	70	94	162	23	12.1	50.3	0.088	0.33	0.73	2100	0.44	61.0
778	75	25	30	1.39	105	75	243	50	12.7	49.4	0.088	0.30	0.78	1556	0.39	58.9
779	75	25	40	2.32	147	63	270	73	15.8	45.6	0.093	0.25	0.79	1450	0.41	58.9
783	50	50	20	0.87	73	84	208	50	9.5	51.6	0.084	0.27	0.66	1655	0.42	61.5
781	50	50	30	1.56	106	68	303	92	13.1	46.3	0.092	0.23	0.67	1330	0.40	60.7
782	50	50	40	2.65	147	56	392	162	18.1	45.3	0.094	0.18	0.65	1052	0.39	58.1
786	25	75	15	0.58	57	98	222	57	8.0	49.9	0.086	0.27	0.57	1790	0.43	62.4
784	25	75	20	0.90	70	78	273	78	7.8	48.7	0.088	0.21	0.53	1268	0.39	60.6
785	25	75	30	1.64	100	61	407	132	11.4	41.1	0.099	0.18	0.54	1050	0.39	61.1
777	0	100	15	0.67	58	86	273	75	2.0	47.6	0.087	0.19	0.56	1085	0.37	60.0
775	0	100	20	0.98	76	78	317	95	3.0	45.6	0.090	0.16	0.50	910	0.37	63.1
776	0	100	30	1.82	105	58	423	163	8.4	41.5	0.096	0.11	0.45	774	0.35	59.9

[a] The spruce contained 50% and the aspen 45% moisture on the wet-wood basis. The proportions of wood in the mixtures are expressed on the moisture-free basis.

[b] An aluminum oxide abrasive, average 90-grit, induced-pore pulpstone was used. It had been dressed with a 10-cut, 1½-inch spiral burr followed by a 14-point diamond burr. The stone had been used 97 hours since the last burring, and prior to the start of this work was judged to be in a medium-sharp condition. Peripheral speed was about 3,470 f.p.m., and grinder-pit temperature and consistency were 165°F. and 2.6 to 3.4%, respectively. Three pockets were in use simultaneously, except in runs 774, 779, and 782, where two pockets were used. Stone immersion was 9 inches.

[c] Basis weight of test sheets, 115 pounds per ream (25 × 40—500).

[d] Total thrust of the pressure foot (determined by calibration of the cylinder pressure) divided by 96 square inches, the area represented by the product of the pocket width and wood length.

[e] Per square foot of wood-stone contact area.

[f] Calculated from amounts retained on 24-, 42-, 80-, and 150-mesh, and passing 150-mesh Tyler screens.

ably be pulped at a faster rate than if the aspen block were replaced with a spruce block. This action gives the equivalent effect of grinding the aspen at a lower pressure and the spruce at a higher pressure than their relative pressures if ground separately at the same rate. A study of the data indicates, however, that this equivalent difference in effective pressures must be small. For example, in considering run 781 on a 50-50 mixture at a grinding pressure of 30 p.s.i. and a grinding rate of 1.56 tons per square foot per day in comparison to the rates at which the two woods are pulped when ground separately, it is estimated that a pressure of about 28 pounds would give this rate on aspen and of about 32 pounds would give it on spruce. Pulps produced at these pressures would not be expected to differ appreciably from those ground at 30 pounds. It would, therefore, be expected that the pulp produced from the 50-50 aspen-spruce mixture of wood would have about the same properties as a mixture of equal parts by weight of spruce and aspen groundwood pulps made separately at the same pressure. Fairly good agreement was found between values for mixtures of pulps and for pulps prepared by grinding the mixed woods. Table II presents some comparative data on two spruce-aspen pulps.

Table II. Comparative Data on Spruce-Aspen Pulps Prepared in Different Ways

Method of preparation	Grinding rate moisture-free wood per 24 hours,[a] tons	Energy consumed,[b] hp.-days	Freeness (Canadian Standard), ml.	Bursting strength	Tensile strength, p.s.i.
50% spruce and 50% aspen ground at 30-pound pressure					
Mixture of pulps (runs 773 and 776)			123	0.22	1193
Calculated from pulps ground separately	1.60	67	109	0.24	1387
Mixture of woods (run 781)	1.56	68	92	0.23	1330
25% spruce and 75% aspen ground at 20-pound pressure					
Mixture of pulps (runs 772 and 775)			68	0.23	1440
Calculated from pulps ground separately	0.91	85	74	0.22	1320
Mixture of woods (run 784)	0.90	78	78	0.21	1268

[a] Per square foot of wood-stone contact area.

[b] Per ton of moisture-free wood ground.

In normal operations, when the two woods are ground separately, the conditions used for spruce would not necessarily be the same as for aspen. From the agreement of calculated and experimental values shown in Table II, it can be postulated that a fair estimate of the properties of a mixture of spruce and aspen groundwood pulps produced under different conditions might be obtained by calculation from the values of the separate pulps.

In this work the moisture content of the wood was kept constant. That of aspen was 45% and that of the rewetted spruce was 50%. It is known that the moisture content of wood affects its grinding characteristics and pulp properties. In other work at the Forest Products Laboratory, it was noted that when aspen was ground at a given pressure, the freeness decreased without much change in the bursting and tensile strengths and grinding rate with decrease in the moisture content of the wood. Therefore it seems that in grinding spruce-aspen mixtures the component pulps would tend to be closer in freeness if the aspen were drier than the spruce.

CONCLUSION

In grinding mixtures of white spruce and quaking aspen, the grinding load is independent of the composition of the mixture. The grinding rate and pulp freeness increase and the energy consumption and pulp strength decrease with the aspen content. All of these relationships are in nearly linear proportions.

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Pulping of Scrub Oak (*Quercus Laevis*) by the Kraft Process

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Turkey (*Quercus laevis*) oak has been investigated as a source of filler pulp for white papers. Pulping conditions studied were combinations of 12, 14, and 16% chemical; 20, 35, and 50 grams per liter liquor concentration; 100 and 115 p.s.i.g. cooking pressure; all at a sulphidity of 25%. Strength characteristics and bleachability of representative samples have been determined. The data show that optimum conditions for producing a pulp requiring 8 to 10% available chlorine are 14 to 16% chemical; 20 to 35 grams per liter liquor concentration; 115 p.s.i.g. cooking pressure; 3 to 3½ hours total cooking time, 1 hour to final temperature. These pulping conditions produce a pulp from turkey oak comparable to other short-fibered bleached pulps used commercially.

THE vast acreages of scrub oak in the South presents a serious problem on the utilization of these hardwoods. It is estimated (*1*) that in Florida alone there are over 5000 square miles of scrub oak forests. The ratio of hardwood to pine forests has continually increased until now slightly over half the forest lands in the South are hardwoods.

Thus far the only practical utilization of turkey (*Quercus laevis*) and blue jack oaks (*Quercus cinerea*) has been as a source of firewood which uses only a small fraction of the total wood available. It was with the thought in mind of using this very large supply of hardwood in the paper industry that this research was undertaken.

During the Second World War, small amounts of oak were used as a diluent to pine in the kraft digesters. Of course, the oak was found to be unsatisfactory because of its extremely short fiber. In addition, it seemed very unlikely that pulping conditions used for cooking pine would be appropriate for oak.

One outstanding characteristic of the oaks which prevented their use in the paper industry is the nature of their bark. Ordinary barking procedures will not remove the bark which constitutes 25% of the log. A method (*2*) has been devised at the Engineering and Industrial Experiment Station of the University of Florida by which the bark and wood may be separated satisfactorily. This method consists of first chipping the logs without removing the bark during which operation the shattering action of the knives separates bark from wood. The mixture of bark and wood chips is then passed over a Sutton, Steele, and Steele air-flotation table which operates on the same principle as the Wilfley table used in the mineral industry for the separation of ores of different specific gravity. An air stream passing up through the bed tends to float the chips and the vibration of the table imparts momentum to them. In addition to the forward movement of the chips they receive a transverse motion depending on the amount of tilt applied to the table. The result is that the wood chips are discharged near the feed end of the table and the bark leaves at the opposite end. The middle fraction, a mixture of bark and wood, is sent back to the feed hopper and repassed over the table.

At no time in this research was any attempt made to produce a fiber which might be used in the kraft process as a replacement for pine. The shortness of the fiber inherent to the oaks made it obvious that it could only be used as a filler pulp similar to the so-called "soda pulps" produced from chestnut, gum, and northern oaks. The work reported by Forman and Niemeyer (*3*) indicated that conditions which might be satisfactory for pine would overcook scrub oak as their unscreened yield was only 39.9%. The semichemical process did not seem feasible since a short-fibered filler pulp should be easy-bleaching if it is to be used in book and bond papers. It was therefore decided to investigate the pulping characteristics of the wood using the sulphate process with less total chemical than is normally employed for producing a bleachable kraft pulp.

The work reported here should be considered as preliminary in nature, as much more experimentation is necessary before the best possible conditions for high yield, bleachability, and strength can be stated definitely. However, from the work done thus far it is apparent that the scrub oaks may be cooked to a satisfactory filler pulp.

METHOD OF PROCEDURE

Logs of turkey oak 5 feet long varying in diameter (with bark on) from 2 to 8 inches were used. Bark was removed by means of a chain debarker rotating at 1350 r.p.m., as the amount of wood used was insufficient to warrant the use of an air-flotation table, the capacity of which is 3000 pound per hour. Chipping was carried out in a No. 0 Mitts and Merrill brush hog. The small size of the feed spout on the hog required cutting of the logs in 1-foot lengths and splitting them so that they would pass through the 4-inch square opening feeding the hog.

Chips were screened on a Day Ro-ball screen and the fraction passing ¾-inch screen opening and retained on No. 4 mesh were used. These chips were air-dried and stored in a well-ventilated bin. The day prior to pulping, sufficient chips for the day's operation were oven-dried and stored in air-tight containers.

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The pulping equipment consists of seven separate digesters in the same steam jacket. Each digester is a No. 316 stainless-steel pipe 6 inches in diameter, reduced at each end by bell reducers to a diameter of 2 inches. The capacity of each digester is $^1/_2$ cubic foot. At one end of the digester is a 2-inch stainless quick-opening gate valve used for blowing the cook. At the other end is a 2-inch union which may be opened for charging of wood chips and cooking liquor. Next to the union is a $^3/_4$-inch valve used for relieving the digester, liquor sampling, and admission of wash water at the end of the cook. The reducing bushing between the 2-inch union and $^3/_4$-inch valve is fitted with a stainless screen which permits liquor sampling without plugging the valve with chips.

The digester housing is a section of 24-inch pipe with flanged ends. Inside this 24-inch pipe are the seven digesters, one in the center with six others surrounding it. The 2-inch portions of the digesters protrude through the flanged ends, with a steam-tight packing gland at each point of leaving the flanges. The digester assembly rotates at 3 r.p.m. to insure thorough mixing of chips and cooking liquor during the cook.

Heating of the digesters is accomplished by admitting steam to the 24-inch digester housing by means of a 1-inch hole through one trunnion. Condensate is removed through a $^1/_2$-inch hole in the other trunnion.

A flexible 2-inch blowline is used to connect any of the digesters to the blowpit. Hot water for washing the digester and blowline after each blow is maintained in a tank that is heated by direct steam to a pressure approximately 15 p.s.i. higher than cooking pressure. A flexible steel hose is used to connect the wash-water tank to the $^3/_4$-inch valve end of any of the digesters just before a blow.

Pulping procedure is as follows. With the 2-inch union end of each digester tube disconnected a weighed portion of dry chips is admitted. Cold cooking liquor of the proper concentration is then poured into the digester and the union is tightened. As many digesters are charged in this manner as are necessary to define the cooking curve for that particular set of cooking conditions.

As soon as all digesters are charged, rotation is started and steam is admitted to the housing through manually operated pressure control valves. These valves are used to bring the digester temperature to the required maximum at a uniform rate in a predetermined time. Throughout this study, the digesters were brought to maximum temperatures in 1 hour.

When a cook is to be blown, the digester housing is stopped and the blowline is connected to the 2-inch quick-opening valve by means of a 2-inch union. The digester is blown by a quarter turn of the quick-opening

Table I. Yield, Strength, and Bleach Data[a]

Cook No.	Active alkali, per cent Na_2O to wood	Original liquor concentration, g. Na_2O/l.	Cooking pressure, p.s.i.g	Total[b] cooking time, hr.	Total yield, per cent of original wood	Screenings, per cent of total yield	Screened yield, per cent of original wood	Beating time to 500 C.S.F., min.	Strength data at 500 Canadian Std. freeness: Breaking length, meters	Tear factor	Burst factor	Double folds	Bleach[c] requirement, per cent available chlorine
1–4	12	50	100	2.5	48.8	3.3	47.2	22	6000	85	28	30	..
1–6	12	50	100	3.5	50.0	3.8	48.1	24	5500	81	25	20	..
2–4	14	50	100	2.5	48.5	1.8	47.7	18	5200	70	24	20	13.8
2–6	14	50	100	3.5	48.7	0.2	48.6	18	5600	79	27	30	13.0
3–3	16	50	100	2.0	47.6	1.3	47.0	10	5600	72	26	25	..
3–4	16	50	100	2.5	46.4	0.6	46.1	10	5700	72	30	22	..
3–5	16	50	100	3.0	46.9	0.3	46.7	15	6000	64	28	10	..
3–6	16	50	100	3.5	46.1	0.1	46.0	..	..	..	..	..	9.5
4–2	12	50	115	1.5	51.7	3.8	49.7	18	6500	77	33	30	..
4–6	12	50	115	3.5	47.3	1.4	46.7	18	6000	77	31	20	..
5–2	14	50	115	1.5	48.8	3.0	47.3	15	5200	69	24	22	..
5–5	14	50	115	3.0	47.4	0.9	47.0	..	..	..	..	..	10.2
5–6	14	50	115	3.5	47.2	0.7	46.9	18	5100	67	25	20	..
6–1	16	50	115	1.0	48.5	1.4	47.8	14	5300	82	25	20	13.1
6–3	16	50	115	2.0	45.7	0.3	45.6	9	5300	68	25	10	8.8
6–6	16	50	115	3.5	44.2	0.0	44.2	8	5300	68	23	10	9.0
7–5	12	35	100	3.0	50.5	4.4	48.3	16	6000	90	31	30	..
7–6	12	35	100	3.5	50.6	3.5	48.8	12	5400	76	27	25	..
8–3	14	35	100	2.0	49.2	2.1	48.2	15	5700	81	29	30	..
8–5	14	35	100	3.0	47.6	0.6	47.3	..	..	..	..	..	9.4
8–6	14	35	100	3.5	47.5	0.7	47.2	16	6100	113	32	40	..
9–2	16	35	100	1.5	49.7	2.8	48.3	17	6000	101	30	40	..
9–6	16	35	100	3.5	47.3	0.0	47.1	18	6100	81	34	42	..
10–4	12	35	115	2.5	51.5	4.3	48.3	12	6100	92	30	50	21.6
10–6	12	35	115	3.5	47.7	3.2	46.2	18	6200	84	31	55	24.0
11–2	14	35	115	1.5	49.9	4.7	47.6	14	5400	76	28	20	..
11–6	14	35	115	3.5	47.3	1.0	46.8	15	5800	80	29	35	..
12–2	16	35	115	1.5	47.5	2.0	46.5	15	5800	84	31	35	..
12–5	16	35	115	3.0	45.7	0.3	45.6	..	..	..	..	..	8.4
12–6	16	35	115	3.5	45.7	0.3	45.6	15	5600	74	29	35	..
13–6	12	20	100	3.5	51.5	10.2	46.2	..	..	..	..	..	..
14–4	14	20	100	2.5	48.5	1.3	47.9	15	5400	71	24	15	..
14–6	14	20	100	3.5	47.3	0.6	47.0	8	6400	68	26	20	13.5
15–3	16	20	100	2.0	49.0	1.6	48.2	15	5800	69	27	20	16.3
15–6	16	20	100	3.5	46.2	0.1	46.2	8	5700	66	28	20	..
16–6	12	20	115	3.5	49.1	13.2	42.6	..	..	..	..	..	10.0
17–3	14	20	115	2.0	48.6	3.6	46.8	12	5600	80	27	20	..
17–6	14	20	115	3.5	46.7	1.1	46.2	12	5800	85	27	45	..
18–3	16	20	115	2.0	47.1	2.4	46.0	9	5000	85	24	15	..
18–4	16	20	115	2.5	44.3	2.0	43.5	..	..	..	..	..	11 2
18–6	16	20	115	3.5	45.0	0.9	44.6	13	5500	85	27	30	10.1

[a] Data are shown only for cooks on which strength and/or bleach tests were run.
[b] All cooks were brought up to full cooking pressure in one hour.
[c] Per cent available chlorine to obtain 75 G.E. brightness.

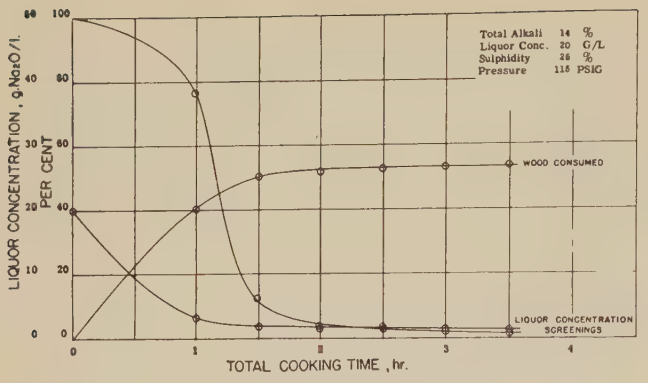

Fig. 1. Cooking Curve

valve. The digester is then charged with wash water and blown again. If necessary, this washing process may be repeated but it was found that one wash cleaned the digester and blow valve completely.

After blowing, the blowline and wash-water line are disconnected from the digester and rotation of the housing is started; this rotation continues until the next cook is to be blown.

The blowpit is then opened and the pulp is washed into a canvas bag and set aside for washing.

A black liquor sample is removed from each digester $2^1/_2$ minutes before the digester is blown. The sample is immediately cooled to room temperature. One hundred milliliters of this sample are mixed with 100 ml. of 10% $BaCl_2$ solution, and the mixture is diluted to 500 ml. in a volumetric flask.

The barium precipitate from all of the black liquors obtained from this investigation exhibited extremely poor settling characteristics. For that reason, rapid separation of precipitate was obtained by centrifuging the samples at 2300 r.p.m. for 10 minutes. The supernatant liquor was drawn off and NaOH and Na_2S were determined, using TAPPI Standard T 625. Na_2S was determined by the H_2S generation procedure and NaOH by use of a Beckman pH meter.

In a few instances it was not possible to titrate the samples on the same day of sampling and centrifuging. In these cases the supernatant liquor from the centrifuge was stored overnight at 5°C. and the analysis made on the next day.

Early pulping experiments with scrub oak proved that wherever the pulp is handled with large quantities of water, such as washing and screening, as much as 10 to 15% of the unscreened yield will be lost through a screen as fine as 120 mesh. For that reason, canvas bags were used for pulp recovery in all washing and screening operations.

Thorough washing is obtained in bags large enough to contain a complete charge from one digester. These bags are hung on a rack in such a manner that the top is kept completely open. They are then washed for several hours until no trace of black liquor can be detected in the drainings. Each bag is then removed from the rack and transferred to a hydraulic press where the pulp is pressed to approximately 75% moisture. The moist pulp is broken up into small crumbs, weighed, and stored overnight in tightly covered 5-gallon cans. The following day moisture samples are taken and total yield is calculated.

The pulp is screened, after total yield has been determined, by using a Valley laboratory flat screen with 0.010-inch slotted plate. The accepted fibers are recovered by collecting them in another canvas bag tied over the discharge of the screen. When screening is complete, the pulp is pressed and moisture determination is carried out as described for the unscreened yield. Screenings are collected, dried in an oven, weighed, and then discarded. The screened pulp is stored for use in physical test sheets and for bleaching tests.

Selected pulps were prepared for physical tests in the following manner. Hydration was developed according to TAPPI Standard T 200 in the $1^1/_2$-pound Valley Niagara beater using 4500 grams on the bed plate. Samples were removed every 15 minutes until a hydration of less than 100 freeness (Canadian Standard) was reached. At each interval, Canadian Standard freeness was determined.

Test sheets were made in accordance with TAPPI Standard T 205 with the exception that a standard Valley sheet mold was used in sheet formation and a Preco hydraulic press was used for wet pressing. Five test sheets were made at each beating time interval of 15 minutes from zero to the point where freeness dropped below 100. Sheets were dried overnight, using drying disks and rings, in the constant temperature room (73° F.—50% humidity).

Since the Valley sheet mold yields a larger test sheet than the TAPPI Standard, $7^3/_4$ inch as compared to $6^1/_4$ inch diameter, it was possible to obtain more test specimens than usual from each sheet.

After conditioning in the constant temperature room, test sheets were tested for weight and thickness before being cut for individual tests. Bursting strength was obtained with the standard Mullen tester; folding endurance with the Schopper folding tester; tear with the Elmendorf tear tester and breaking length with a Thwing-Albert electrohydraulic tensile tester. All strength data were calculated in the units prescribed by TAPPI Standard T 220 m.

Selected pulp samples were tested for bleach requirements in accordance with TAPPI Standard T 219 m. Handsheets for optical test were formed according to TAPPI Standard T 218 m. Brightness of test specimens was obtained as specified by TAPPI Standard T 217 m using the G.E. brightness tester.

PRESENTATION OF DATA

Preliminary investigations showed that if more than 16% total chemical (grams Na_2O per 100 grams wood)

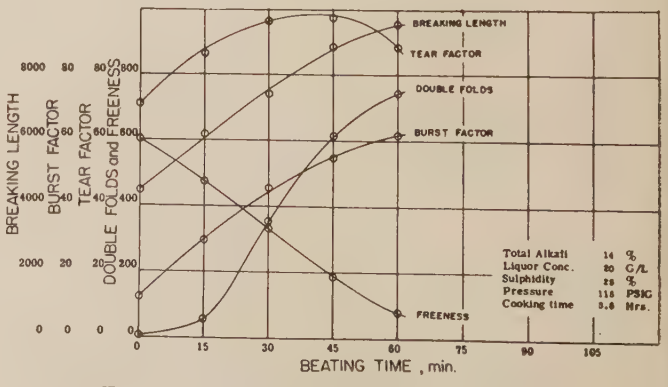

Fig. 2. Physical Characteristics of Pulps

were used, very low yields were obtained while if 10% total chemical were used, the pulps were extremely raw. For that reason, the per cents total chemical studied were 12, 14, and 16. At each of these values, chemical concentrations of 20, 35, and 50 grams per liter, expressed as Na_2O, were studied. All of these cooks were carried out at both 100 and 115 pounds steam pressure (gage). Thus far only one sulphidity has been investigated, namely 25%.

For each set of cooking conditions black liquor concentration, total yield, screenings, and screened yield were determined. Lignin content was determined for pulps of various yields. Selected pulp samples, which showed reasonable screened yield, were used for physical testing. With the exception of the cooks of series 13 and 16, at least two samples of each series were used for physical testing. In general, they were the cooks of shortest and longest cooking time that also had less than 5% screenings. The bleachability of 16 pulp samples selected to cover, as well as possible, the whole range of pulping conditions was determined.

In Table I are presented yield, strength, and bleach data for only those cooks on which strength tests and/or bleach tests were run. The strength data are limited to values of breaking length, tear factor, burst factor, and double folds at 500 freeness (Canadian Standard). The length of beating time to develop this freeness is also shown. The bleach requirement is presented as per cent available chlorine to produce a G.E. brightness of 75.

The cooking data were plotted in the form of the curves of Fig. 1 which shows the relationships of wood consumed, chemical concentration, and per cent screenings (based on total yield) to cooking time.

The strength data were plotted against beating time as shown in Fig. 2. The particular chart shown here represents one of the pulps of best all-round strength development. Several other pulping conditions gave comparable strength development.

The relationship between per cent screened yield and wood consumed is shown in Fig. 3. Figure 4 shows the rate of chemical consumption as wood is consumed.

The lignin content at various total yields is shown in Table II. Figure 5 is a correlation between per cent lignin removed and per cent total wood consumed.

As has been mentioned previously, exploratory cooks showed that the range between 12 and 16% chemical, expressed as grams Na_2O per 100 grams wood, showed

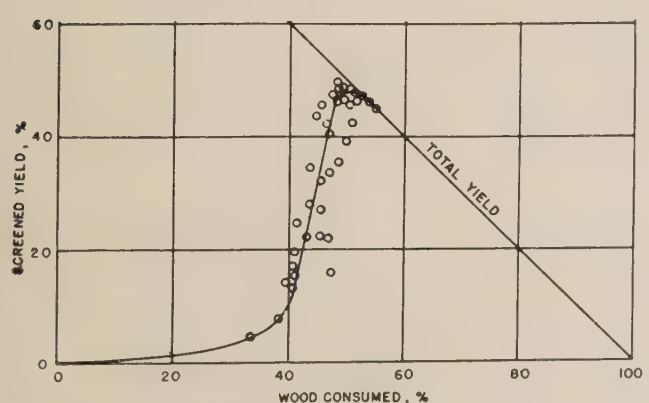

Fig. 3. Per cent screened yield vs. per cent wood consumed

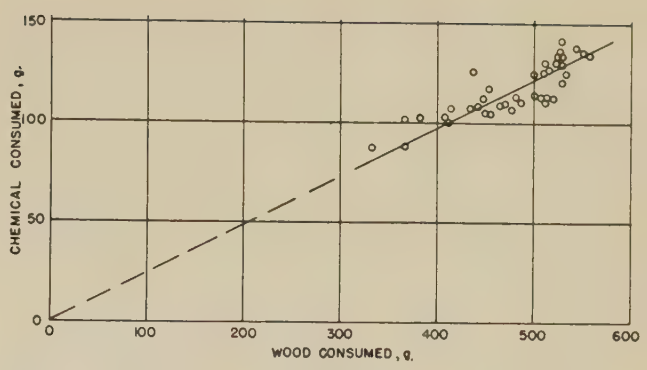

Fig. 4. Grams chemical consumed vs. grams wood consumed

the best possibility of producing a bleachable pulp of reasonable yield. When 18% chemical is used, very low yields, 40% or under, are realized and 10% chemical produces almost 100% screenings. Ratio of sulphidity to total alkali (expressed as Na_2O) was held at 25%, not because it is thought that 25% sulphidity is best suited for pulping scrub oak, but because one single sulphidity had to be stipulated for this preliminary exploratory work.

Cooking curves, as shown in Fig. 1, were drawn for each set of conditions of per cent chemical, liquor concentration, and cooking pressure. These curves were compared by superimposing one on the other over a strong light. It would be very difficult to draw any conclusions, other than those generally known, as to the effects of changes in per cent chemical, liquor concentration, and cooking pressure on the rate of pulping. This is due to the very narrow range in which this wood may be pulped and still obtain the product desired. The maximum difference in per cent wood removed at $3^1/_2$ hours total cooking time is only $6^1/_2$% from the rawest pulp (51.5% total yield, 10.2% screenings) to the one most thoroughly cooked (45.0% total yield, 0.9% screenings). In general, per cent chemical controls the ultimate yield; concentration and pressure, in the range studied, control the speed at which that yield is approached.

An examination of the curves for per cent screenings vs. cooking time for each set of cooking conditions shows, in some cases, that at early stages of the cook, for any given per cent chemical, decreases in initial liquor concentration produce decreases in per cent screenings without necessarily affecting the total yield

Table II. Lignin Content of Pulp[a]

Cook No.	*Active alkali, per cent Na_2O in wood*	*Original liquor concentration, g. Na_2O/l.*	*Cooking pressure, p.s.i.g.*	*Total[b] cooking time, hr.*	*Lignin in pulp, %*	*Total yield, per cent of original wood*
1–1	12.0	50.0	100	1.0	15.0	58.9
1–2	12.0	50.0	100	1.5	7.3	53.0
1–3	12.0	50.0	100	2.0	3.3	51.3
1–4	12.0	50.0	100	2.5	3.5	48.8
1–5	12.0	50.0	100	3.0	3.3	49.3
1–6	12.0	50.0	100	3.5	2.5	50.0
2–6	14.0	50.0	100	3.5	2.2	48.7
4–1	12.0	50.0	115	1.0	10.0	55.3
4–2	12.0	50.0	115	1.5	7.3	51.7
4–6	12.0	50.0	115	3.5	3.3	47.3
9–2	16.0	35.0	100	1.5	3.2	47.7
10–4	12.0	35.0	115	2.5	3.9	51.5

[a] Lignin content of wood was 25.3%.
[b] All cooks brought up to full cooking pressure in one hour.

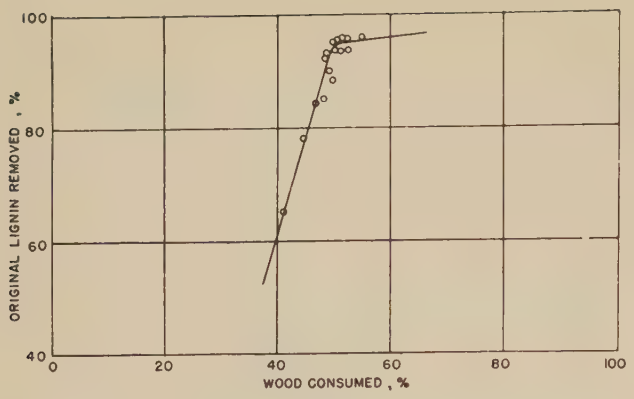

Fig. 5. Original lignin removed vs. per cent wood consumed

at that time. For example, cook No. 7 (12% chemical, 35 grams per liter, and 100 p.s.i.) had the same total yield at 1½ hours cooking time as cook No. 1 at the same cooking time (12% chemical, 50 grams per liter, and 100 p.s.i.). However, cook No. 7 had 34.5% less screenings than cook No. 1. No particular significance can be attributed to this fact. The screening data in the early stages of the cook are not very reliable due to the fact that the screenings of the raw pulps are soft chips or pieces of chips. A slight difference in mechanical handling of the pulps before screening may break down portions of these chip fragments into partially cooked fiber bundles which pass through the 0.01 inch-screen plate slots.

Care should therefore be taken in the handling of the pulps prior to screening. It has been found that the freshly washed pulps from the blowpit should not be pressed to too low a moisture content for yield determinations. These short fibered pulps, when pressed to 65% moisture or less, tend to form small balls of cooked fiber which, unless disintegrated, will appear as screenings. It is quite probable that this disintegration will also break up part of the true screenings sufficiently to cause them to appear in the screened pulp. As soon as this condition was observed in the course of these experiments, pulps were not pressed beyond 20 to 25% dry fiber.

In Fig. 3, a relationship between per cent yield vs. per cent wood consumed is shown. The curve defined by the data points is the screened yield. This curve indicates that the maximum screened yield occurs at 50% wood consumed. Up to 50% wood consumed screenings are excessive, while beyond 50% wood consumed any attempt to reduce screenings results in a decrease in screened yield. However, this maximum of screened yield does not indicate in any way at what point in the cook maximum strength may be developed.

A plot of grams chemical consumed vs. grams wood consumed is shown in Fig. 4. A line drawn through the origin seems to fit the scattered data points but the data are confined to such a narrow region that it is impossible to say positively that this is true.

The same correlation between lignin removed and wood consumed reported by several investigators has been found to be true in the cooks reported here as shown in Fig. 5. The unfortunate thing about all such curves reported is that the break in the straight line occurs at the most important point in the pulping reaction. The use of this relationship as a substitute for actual lignin determinations in the range of 50 to 60% wood consumed does not appear warranted.

Physical Strength of Pulps

The data in Table I and the typical strength curves of Fig. 2 show the physical strength characteristics of scrub oak pulps. The rate at which physical strength develops with beating time varies, depending on cooking conditions. Folding strength was a negligible quantity until after 15 minutes beating time, or a freeness of about 500. After this point, fold developed at a high rate up through the maximum beating time where double folds reached values between 500 and 1000.

Tear factor generally reached a peak value between 30 and 45 minutes beating time, after which the values either leveled off or decreased. In a few cases maximum tear had not been reached after 60 minutes beating time. Burst factor and breaking length increased in almost a straight line throughout the beating cycle.

The strength values at 500 freeness for all pulps as shown in Table I were read from the strength curves. They are presented here to show what strength may be expected from the pulps with a practical amount of hydration in commercial practice.

Bleachability of Pulps

Sixteen samples of pulp were picked from the group to represent the entire range of cooking conditions. Half of the pulps tested were found to bleach to a G.E. brightness of 75% with 10% or less available chlorine. The best of these was cook No. 12–5 (16% chemical, 35 grams per liter, 115 p.s.i., and 3 hours total cooking time), which attained a G.E. brightness of 75% with 8.4% available chlorine. The pulp most difficult to bleach was found to be 10–4 (12% chemical, 35 grams per liter, 115 p.s.i. and 2½ hours total cooking time). As would be expected, this same pulp had the highest physical strength of all pulps tested. Its high yield of 51.5% and high lignin content of 3.9% accounts for its poor bleaching qualities.

Permanganate numbers for the sixteen pulps were determined but the data points do not fall sufficiently close to a straight line when plotted against per cent available chlorine to warrant their use as a substitute for the bleach test. They can be used only as a qualitative test for degree of pulping. For that reason these data are not included in this report.

The data presented in this report do not constitute the complete investigation of scrub oak pulping. Since the optimum conditions for pulping oak with a sulphidity of 25% have been determined, it is now possible to study the effect of changes in sulphidity on these optimum conditions. This is the next major phase of the investigation to be studied.

CONCLUSIONS

From an analysis of cooking, physical strength, and yield data, the data indicate that the best pulping conditions for scrub oak at a sulphidity of 25% are 14 to 16% chemical, 20 to 35 grams per liter liquor concentrations, 115 p.s.i.g. steam pressure, and 3 to 3½ hours total cooking time. A 12% chemical will produce a pulp of high yield and high strength but the bleaching qualities of the pulp are too poor for commercial use. It would be possible to use a liquor concen-

tration of 50 grams per liter but 100 p.s.i.g. should be used instead of 115 p.s.i.g. The more dilute solutions tend to produce a better pulp due to the volume of liquor involved. However, this increased volume has an adverse effect on the economy of the liquor recovery system in the mill. Therefore, the actual commercial concentration must be a balance between high quality pulp and expense of liquor recovery. It appears that liquors of about 35 grams per liter should be the best compromise.

The above conclusions are only applicable at a sulphidity of 25%. Future study will determine whether these conditions are still the best at other values of sulphidity. At any rate, the conditions specified will produce a high quality filler pulp from turkey (*Quercus laevis*) oak for white papers.

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Modulus of Rupture Test for Paraffin Wax

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The modulus of rupture of paraffin wax is determined by breaking small prepared slabs of paraffin by applying a load midway between two supports. The load is controlled by flowing water into a plastic bucket suspended from the ends of the loading bar that rests on the slab of wax. The paraffin slabs are prepared by casting over water. Equipment for performing this test is commercially available. Data showing the stiffening effect of paraffin on paper and the function of the modulus of rupture on this effect are presented as well as evidence of the reproducibility of the test.

The modulus of rupture test was developed in connection with the control of quality of paraffined fiber milk containers. Previous to the adoption of this test by the American Can Co. in 1944 the fully refined paraffin wax for milk containers was purchased on specifications that referred only to: cleanliness, color, melting point, odor, and taste. It was then discovered that the strength of the wax, as measured by this test, exerted a profound effect on the structural strength and, therefore, the quality of the fiber milk containers. This refers especially to their ability to withstand rough handling and to resist damage in shipment. This effect on the rigidity of the container is in addition to the obvious function of the wax in preventing passage of liquid into the fiber walls of the containers.

The following data illustrate the effect of paraffin wax impregnation on the measured stiffness of paper stock similar to that used in fiber milk containers. In this experiment the stiffness of 0.017-inch cylinder stock was determined on a Taber V5 stiffness tester both before and after dipping in paraffin. It can be concluded that the paraffin contributed appreciably to the stiffness of the paper and that this increase was greater when wax having the higher modulus of rupture was used.

METHOD

Reduced to its fundamentals, the modulus of rupture test consists of breaking a small slab of paraffin midway between two supports. The actual dimensions of the slab at the point of break are determined and the modulus calculated by substitution in the formula:

$$S_R = \frac{3}{2}\frac{Pl}{bt^2}$$

Where

S_R is modulus of rupture
P is the breaking load in pounds
l is the distance between the supports in inches
b is the width of the slab
t is the thickness of the slab.

In the commercially available apparatus l is 2 inches and W is usually weighed in grams. The formula then reduces to:

$$S_R = \frac{0.0066\ W\ (\text{grams})}{bt^2}$$

Figure 3 shows two views of the apparatus that was designed and manufactured for use in the various American Can Co. plants producing fiber milk containers and for several of the suppliers of paraffin wax. It has been installed in American Can Co. plants at Jersey City, Los Angeles, San Francisco, and Maywood, Ill., where every shipment of paraffin for use on fiber milk containers is tested on receipt. The method of procedure was standardized and has been in continuous use in its present form since January, 1945.

The apparatus is somewhat more elaborate than the simple requirements of the test would indicate. These refinements are all designed to make the operation of the equipment as automatic as possible, a desirable feature in view of the large number of shipments to be tested and the fact that it has to be operated by technicians of varied experience. It functions by applying a load at a constant rate to a bar resting on the paraffin slab. The means of doing this is to flow water through an orifice into a plastic bucket suspended from the ends of the bar. When the paraffin slab breaks, the falling bucket automatically causes the stream of water to be diverted into the drain.

The apparatus consists of a tank A mounted on a stand with overflow pipe B to maintain constant head at the orifice F. The overflow drains off at C into the drain D. Water flows from the opening E through orifice F into plastic bucket G. This bucket is con-

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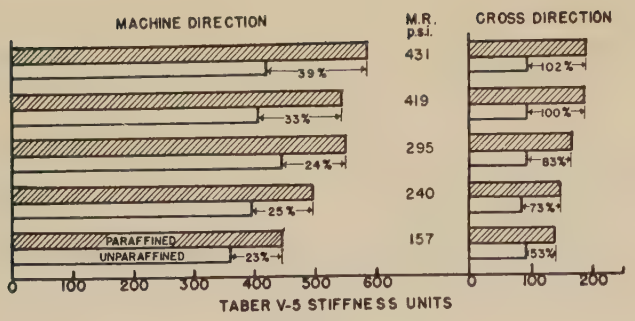

Fig. 1

nected by means of a lightweight spool and aluminum strip to the breaking bar H resting on the parafin slab J which in turn rests on two fulcra K. A gaging lever L is used to properly locate the breaking bar in position. The trip table M is connected through release latches to the water stop N which returns the excess water to sump C on breaking the paraffin slab.

The slabs of paraffin are prepared by melting the wax over hot distilled water in aluminum pans, and allowing solidification to occur slowly. Aluminum pans of suitable size are included with the supplied equipment $8^1/_2$ by 5 by 3 inches. The paraffin in the form of small chips is placed in the pan and about 1 liter of hot-boiled distilled water is added. The paraffin melts and floats on the water. The pan is covered with a glass plate and a hood is placed over it to avoid the effect of drafts.

The weight of the paraffin sample (about 90 grams) is varied slightly depending on the exact dimensions of the pan so that the resulting slab is as near 0.150 inch thick as possible. It was found by experiment that the thickness factor in the formula will not correct for large variations in the thickness of the specimen. The thickness should be 0.150 ± 0.005 inch.

After standing a minimum of 4 hours the solid paraffin slab can be removed from the pan and the edges trimmed off to leave a piece about 9 by 3 inches. This is then cut into $^1/_2$-inch slabs by means of a bench saw also supplied with the equipment (see Fig. 4).

The test pieces should be placed on a glass plate and allowed to stand about 1 hour before testing. The melting, solidification, cutting, and breaking of the test pieces should all be done in a room held at 73 °F. ± 1 °F.

Adjust the rate of flow of water into tank A so that a small amount continues to flow out of the overflow B while the controlled amount is flowing from orifice F. Breaking bar H is lifted by the gaging lever L and the paraffin slab inserted across fulcra K against

Table I

	Breaking weight, g.	*Total weight, g.*	*Thickness, in.*	*Width, in.*	*MR-73°F., p.s.i.*	*Side av., p.s.i.*	*Total av., p.s.i.*
Water side down	860	920	0.146	0.520	548	553	563
	920	980	0.147	0.519	576		
	850	910	0.149	0.512	528		
	930	990	0.148	0.519	574		
	870	930	0.146	0.516	557		
	820	880	0.145	0.518	534		
Air side down	880	940	0.147	0.518	554	573	
	980	1040	0.147	0.520	609		
	940	1000	0.150	0.518	567		
	950	1010	0.148	0.517	588		
	840	900	0.148	0.519	522		
	930	990	0.145	0.518	599		

Table II. Inter-Laboratory Test

Laboratory No.	*Sample No., p.s.i. 1*	*2*	*3*	*4*
1	188	416	264	348
2	170	408	247	314
3	171	415	246	323
4	196	417	292	344
5	175	442	269	322
6	190	434	263	365
7	209	447	302	363

the back gages. The lever is lowered allowing the breaking bar H to rest on the paraffin specimen. This, with the empty bucket and suspension, has been adjusted to provide an initial load of 60 grams. During this operation, the water stop N should be under the orifice so that all water flowing out of the orifice goes into the sump.

With the sample in place, handle O is moved quickly in a clockwise direction as far as it will go. This causes the water to flow into the bucket G increasing the load on the specimen at a constant rate (18 ounces per minute) until the breaking of the sample causes the bucket to fall on M. This in turn automatically moves the water stop under the orifice thus discontinuing the flow into the bucket. The weight or volume of the water (grams or milliliters) plus 60 grams are taken as the breaking load of the specimen.

Both halves of the broken specimen are removed, and width and thickness measurements are taken on both broken ends using a bench comparator. From these data the modulus of rupture is calculated in accordance with the formula given above.

A typical calculation is shown in Table I to illustrate one of the variables encountered in this method.

This example is shown to illustrate the fact that in general there is a significant difference in the strength of the paraffin slab when broken air side down vs. water side down. For this reason the final modulus result represents the average of an equal number of samples broken each way. The cause of this variation is not known with certainty but is assumed to be associated with the different rates of cooling of the two sides of the paraffin slab.

During the development work on this test other methods of casting the slabs were tried and discarded. It was considered that when paraffin is used commercially in the way it is on fiber milk containers, it solidifies in contact with air and, therefore, if a variable exists, it is probably desirable that it be allowed to influence the test results.

Methods of casting in contact with lubricated glass or molds are subject to the objection that many samples become damaged or strained on removal from the contacting surface.

On the basis of the rather extensive experience with this test, it has been customary to break twelve specimens for any given test—six air side down and six

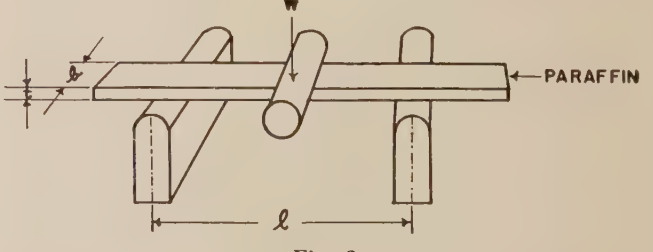

Fig. 2

Table III. Ten Castings of Same Sample

Determination No.	*Water side down p.s.i.*	*Air side down, p.s.i.*	*Average, p.s.i.*
1	338	288	313
2	322	290	306
3	324	283	303
4	327	295	311
5	326	304	315
6	327	283	305
7	332	282	307
8	317	285	301
9	339	289	313
10	343	297	320

water side down. It is not necessary or desirable to allow the operator to discard individual determinations that appear to him to be out of line.

In November of 1945, a rather extensive inter-laboratory test was run on four different samples of paraffin. By this time, in addition to the four American Can Co. laboratories, three other laboratories had been equipped to make modulus of rupture determinations. Identical samples of four lots of paraffin were sent to the seven laboratories with the request that they run duplicate determinations on each of the samples and report the results. Four of the laboratories reported duplicate figures on each of the samples, the other three reported but one determination.

Table II lists the results of this inter-laboratory check on the accuracy of the method. Laboratories Nos. 1 to 4 reported duplicate determinations on each of the samples. The figures in this table are the actual modulus of rupture results in pounds per square inch as reported.

If all of the results in Table II are given equal weight, the standard deviation for each sample is as follows:

Sample	*Standard deviation, p.s.i.*
1	14
2	15
3	21
4	21

$$\text{Standard Deviation} = \sqrt{\frac{(n_1 - a)^2 + (n_2 - a)^2}{n - 1}} \text{—etc.}$$

Where n, n_2, n etc. are the values for a given sample; a = average for the sample.

The inter-laboratory test presented above is admittedly as much a test of the diligence of the various operators as of the true accuracy of the test itself. In order to obtain more information on the reproducibility of the

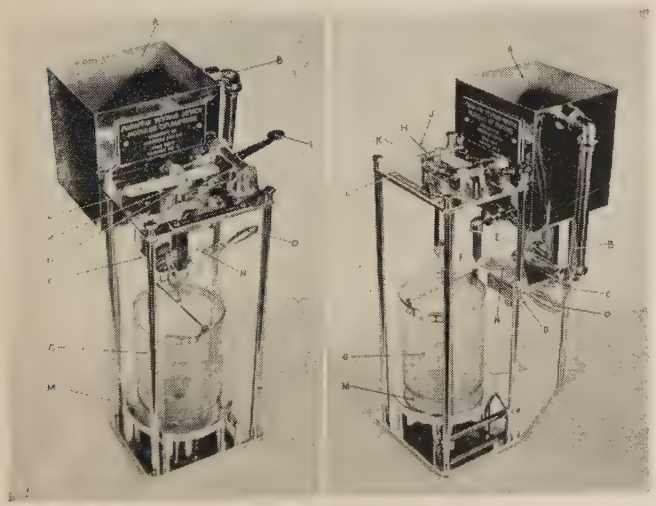

Fig. 3

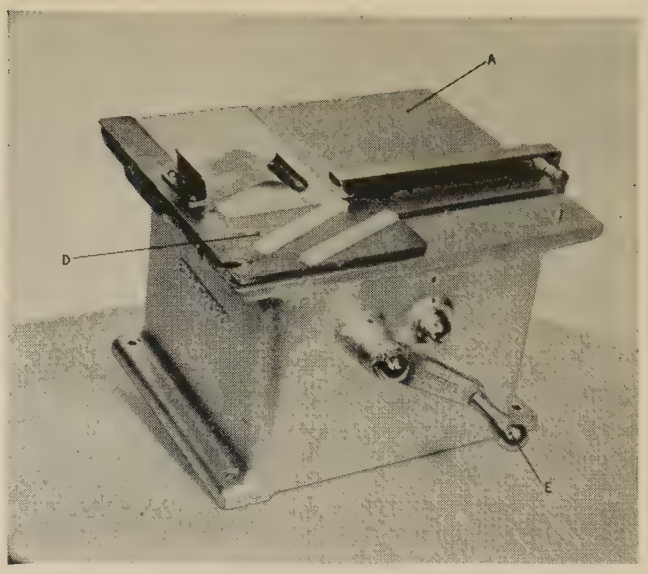

Fig. 4

method, a series of ten determinations, all based on the same paraffin sample, was run. Ten separate castings were made and the modulus determined on a single instrument. Table III gives the results which were obtained.

A calculation based on the ten averages given in Table III, column 3, yields a standard deviation of 6 p.s.i.

The extent to which the individual readings vary can be obtained by considering the standard deviations of the six values used in determining the modulus on a given casting either water side down or air side down. The following apply to the figures in Table III.

Casting No.	*Standard deviations, p.s.i.* *Water side down*	*Air side down*
1	10	10
2	16	7
3	12	9
4	12	9
5	13	8
6	9	10
7	15	13
8	18	7
9	22	13
10	15	10

These values might have been lower had the operator been allowed to reject figures obviously out of line but in this experiment the no-rejection rule was adhered to rigidly.

There was also some evidence in this calculation that the values determined air side down showed less variation than the water side down figures. This observation might not apply over a large number of paraffin samples. It is quite apparent on inspecting data on a large number of samples that the difference between the water side down average and the air side down varies both in magnitude and sign. The reason for this variation has not become apparent and until it is established the best procedure is to report the average of both figures.

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The data presented in Fig. 1 and Table III were obtained by R. L. Skiles and J. R. Wright of the Research Division, American Can Co., D. C. Foord also collaborated with the author in the development of the test. The apparatus is commercially available from Thwing-Albert Co., Philadelphia, Pa.

Some Further Observations on *Penicillium Roqueforti* Thom

JOHN W. APPLING, STANLEY J. BUCKMAN, and CONAN D. CASH

An unusually interesting case of inhibition of mold growth under laboratory conditions was noted in groundwood pulp treated with different amounts of a 10% solution of phenylmercuric acetate. Pulp specimens given the lowest treatment were essentially free of mold growth when incubated in Mason jars while mold growth occurred to an appreciable extent on pulp specimens given three higher treatments with the same toxicant. Antibiosis was suspected as the explanation for the inhibition of mold growth on the pulp given the lowest treatment. The antagonistic organisms were believed to have been inhibited by the higher concentrations of toxicant thus allowing the mold to grow. The mold present was primarily *Penicillium roqueforti* and the authors had previously demonstrated the high resistance of this organism to phenylmercuric acetate. Isolations from the pulp given the lowest amount of treatment confirmed the presence of a rod-shaped, chromogenic, Gram-negative, nonspore-forming bacterium which showed marked antagonism toward *Penicillium roqueforti* under various conditions of growth, such as in malt agar medium and in groundwood pulp to which maltose and sodium nitrate had been added. As previously reported by the authors, limited growth of *Penicillium roqueforti* apparently does not appreciably affect the usefulness of groundwood pulp. However, this mold organism seems to be widely distributed and the observed results thus add to the increasing amount of information available on biological competition between members of the microflora of pulp and paper mills.

THE occurrence of *Penicillium roqueforti* Thom in pulp mills has been reported by various workers during the past few years (*1*, *2*). The opinion has been advanced that many of the *Penicillium* sp. reported by numerous authors as present in pulp and slime samples could have been the species *roqueforti*. Appling, Buckman, and Meals (*1*) demonstrated an unusually high tolerance of this species to phenylmercuric acetate but not to sodium pentachlorophenate. During 1949 a case of unusual moldy groundwood lap came to the attention of the present authors. Mason jar cultures of groundwood lap stock treated with various amounts of phenylmercuric acetate and sprayed with a mixed culture of bacteria and fungi gave rise to a paradoxical situation in which mold growth did not occur on pulp treated with the smallest amount of toxicant but it did occur at three significantly larger amounts of treatment. The authors suspected the observed result might be caused by biological competition in the pulp given the smallest amount of treatment. This paper will present the evidence obtained in support of the above hypothesis.

JOHN W. APPLING, Member TAPPI, Director of Microbiological Research; STANLEY J. BUCKMAN, Member TAPPI, President; and CONAN D. CASH, Bacteriologist, Buckman Laboratories, Inc., Memphis, Tenn.

It will be well to review briefly the work of several Swedish investigators in the field of biological competition as applied to pulp mill systems. In 1933 and 1934 Melin (*4*) as well as Melin and Nannfeldt (*5*) suggested the possibility of adding antagonistic organisms to the groundwood pulp as a means of controlling blue-staining fungi. The action of the antagonistic organisms was attributed to the secretion of growth-retarding and growth-suppressing substances which experiments had shown were not heat stable. Quite recently Gadd (*3*) reported the results of some experiments indicating that antibiotic poisons may not be the primary competitive factor under some conditions. He offered evidence which indicated that the antagonism was the result of competition by microorganisms for nutritive substances in groundwood pulp which is normally quite low in nitrogen and phosphorus. Pehrson (*6*), also of Sweden, recently isolated from fresh groundwood pulp and white water a species of bacterium which has strong fungistatic properties. He observed that this species was abundant in fresh groundwood pulp, in white water, and in slime from various Swedish mills. He also found that the antibiotic substance produced by the bacterium was heat stable.

PRELIMINARY INDICATIONS OF BIOLOGICAL COMPETITION

Four test lots of groundwood pulp were treated at Hantsport, Nova Scotia by adding 0.52, 0.73, 0.98 and 1.43 pounds of BSM-11B, a 10% solution of phenylmercuric acetate, per ton of wet pulp to the system while the pulp was being produced. Analyses of samples of pulp from the different lots indicated that between 40 to 65% of the phenylmercuric acetate added to the system was present in the wet pulp. When the pulp from the different lots was received at Chester, Pa., two sets of strips were cut from each lot and placed in closed Mason jars. The jars containing one set of specimens from each lot were sprayed with a mixed culture of bacteria and fungi to supplement the inoculum present in the pulp while the jars containing the other set of specimens were not given a supplementary inoculation. Both sets of specimens were incubated in closed Mason jars from April 8 to Sept. 22, 1949. Mold growth developed on the pulp in all cases except that given the smallest amount of treatment. Figure 1 is a photograph of representative strips from the Mason jars which contained sheets treated with the smallest amount (see top row) and with the next larger amount (see bottom row).

The predominant mold present on the pulp specimens shown in Fig. 1 has the characteristics of *P. roqueforti*, including a typical violet stain on some of the pulp and having the odor of roquefort cheese. It

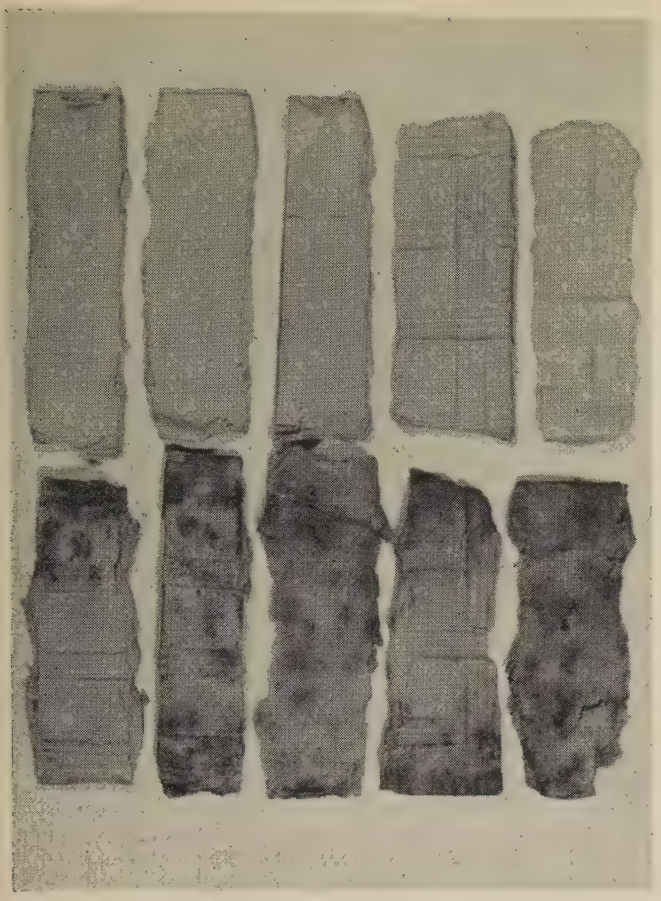

Fig. 1. Condition of treated specimens of wet groundwood pulp sprayed with a mixed culture of bacteria and fungi and incubated at room temperature in closed Mason jars at Chester, Pa. from April 8 to Sept. 22, 1949

The specimens in the top row received the smaller treatment with phenylmercuric acetate while those in the bottom row received the larger treatment.

was postulated that the smallest treatment was inadequate to inhibit the growth of other microorganisms in the pulp which competed with *P. roqueforti* and inhibited the growth of this organism, probably by the secretion of antibiotics which exerted a fungistatic effect. Evidence of the growth of other organisms was indicated by a pressure in the culture jar when it was opened and by a typical "sour" fermentation odor. This was distinctly different from the odor of the opened jars containing pulps treated with three progressively larger amounts. The odor of the latter jars when first opened was that of roquefort cheese but this changed somewhat to a typical "musty" odor. The dominant odor was clearly that produced by *P. roqueforti*. It is believed that this mold grew particularly well on the pulp containing the higher concentrations of phenylmercuric acetate because competing microorganisms had been eliminated by the phenylmercuric acetate which is relatively low in effect against *P. roqueforti* (*1*).

The occurrence of *P. roqueforti* in groundwood pulp under mill conditions of storage has been amply demonstrated (*2*); however, its presence is more usually associated with light violet staining, particularly around the outer edges of pulp laps and sheets. Normally, this mold does not show an extensive development of green fruiting bodies on pulp in bales. Nevertheless, its presence has been confirmed and its limited effect upon pulp quality has been described in an earlier paper (*1*).

CONFIRMATION OF BIOLOGICAL COMPETITION

In order to test the hypothesis which was advanced to explain why no growth of mold had occurred on groundwood lap given the smallest amount of treatment with phenylmercuric acetate whereas abundant growth occurred at three larger amounts of treatment with the same toxicant, it was necessary to isolate from the pulp receiving the smallest treatment, microorganisms which were definitely and strongly antagonistic to *P. roqueforti*. This proved to be more difficult than the authors had anticipated and it is believed that a brief description of the isolation of antagonistic microorganisms will be of general interest to those concerned with mold prevention in groundwood pulp.

The first attempt to isolate antagonistic microorganisms was made by placing some of the groundwood pulp under investigation on malt and Bacto nutrient agar plates. Profuse growth of a greenish-yellow mold occurred on the malt agar plates which contained bits of pulp as inoculum. Sparse growth of white and greenish-yellow colored colonies of bacteria was observed on the plates which had been streaked with a suspension of the pulp. A curious thing occurred in the Mason jar and in the Petri dish which contained the main portion of the pulp. Profuse growth of a greenish-yellow mold, which seemed the same as that already observed on the malt agar plates, occurred in both instances.

The next attempt to isolate the antagonistic microorganisms was initiated with the incubation of a series of 20 flasks containing suspensions of the groundwood pulp in question for 24 to 48 hours at 28°C. The purpose of the incubation period was to encourage the growth of bacteria which the first attempt at isolation had shown to be present in small numbers. Ten of the flasks were sampled after about 24 hours and streak plates were made using both malt and nutrient agars. After about 48 hours the remaining ten flasks were sampled in the same manner. Excellent growth resulted on most of the plates irrespective of the flasks used. In general both white and greenish-yellow colonies appeared on all plates. Some 30 well-isolated colonies were picked for subculture and subsequent study. Greenish-yellow colonies predominated on the nutrient agar plates and white ones on the malt agar plates. When a colony was picked on nutrient agar,

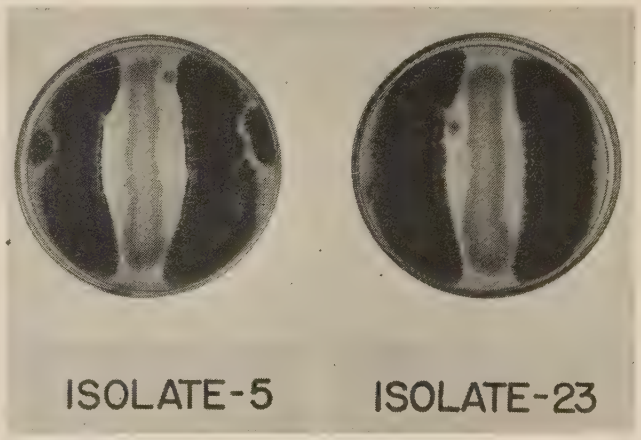

Fig. 2. Antagonism between bacterial isolates from groundwood pulp and Penicillium roqueforti *Thom, when parallel inoculations were made on the surface of malt agar*

it was transferred to nutrient agar slants and similarly colonies on malt agar were transferred to malt agar slants. All cultures were incubated at 28°C.

The next step was to test a representative number of both greenish-yellow and white subcultures for their fungistatic properties. From the microscopic examination of stained smears from the isolated organisms, it appeared that little difference existed among the 30 isolates. All seemed to be nonspore-forming, short rods producing greenish-yellow pigment on nutrient agar but not on malt agar. Because of the morphological similarity between these isolates, only seven were selected for fungistatic tests. *P. roqueforti* was naturally selected as the test organism to check antagonism. The first tests were made in various ways but the best results were obtained when a central streak of the bacterial isolate on malt agar plates was flanked on both sides by approximately parallel streaks of *P. roqueforti*. Not all of the isolates inhibited this mold during subsequent incubation at 28°C. Isolates 5 and 23 were finally selected for additional tests using both groundwood pulp and malt agar as substrates. Isolate 5 grew equally well on malt and nutrient agar slants, producing the greenish-yellow pigment on the latter but not on the former.

Using malt agar as the medium suitable for both bacterial isolates and *P. roqueforti*, the authors obtained many results similar to the plates shown in Figs. 2 and 3. These plates were photographed after 14 and 5 days, respectively, of incubation at 28°C. Those in Fig. 2 show clearly the fungistatic effect of both isolates 5 and 23 when the streaks were made on the same surface. In the case of Fig. 3, isolates 5 and 23 were introduced in only one half of the agar, namely, that shown in the lower half of the plates. *P. roqueforti* was streaked across the entire plates but it failed to grow in the lower halves which contained the bacterial isolates.

Spruce groundwood pulp enriched by the addition of maltose and sodium nitrate was also used to demonstrate the fungistatic properties of isolates 5 and 23. Mats of pulp containing about 6 grams of fiber were made as described by Appling, Buckman, and Meals (*1*) except that no adjustment of moisture content or weight was made in the present study. A sufficient amount of a

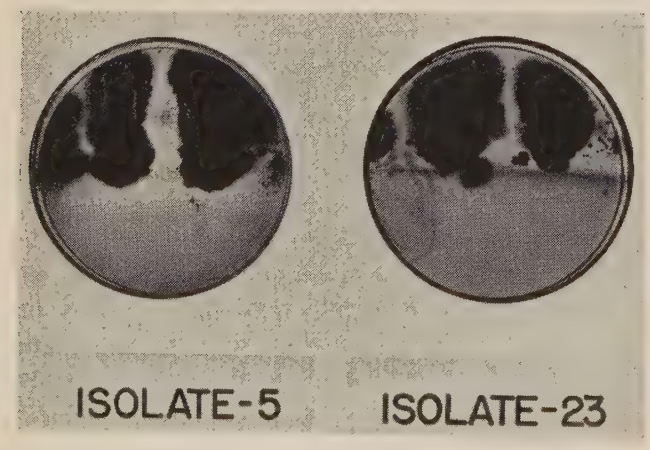

***Fig. 3. Antagonism between bacterial isolates from groundwood pulp and* Penicillium roque forti *Thom when one half of the malt agar plates were inoculated with the bacterial isolates followed by streaking across the plates with* P. roqueforti

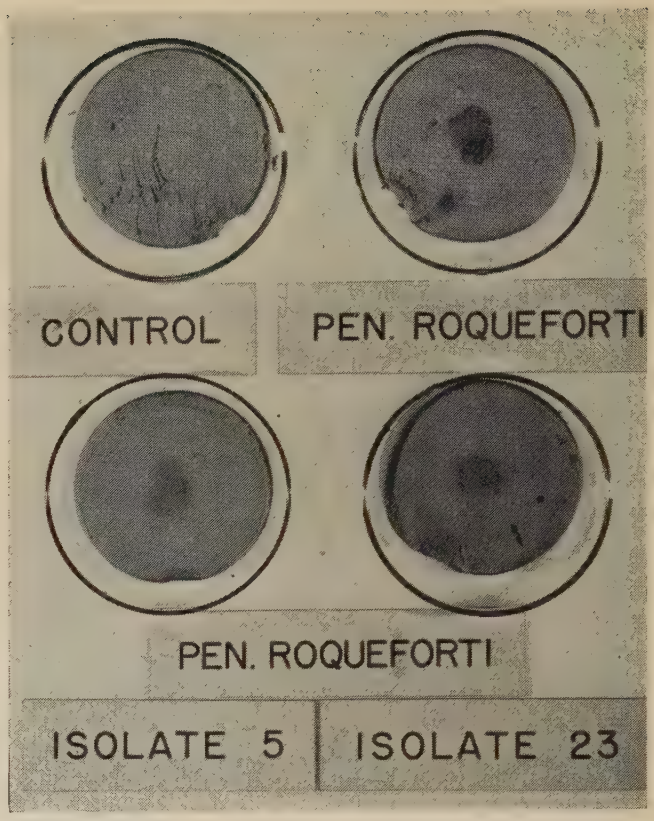

Fig. 4. Antagonism between bacterial isolates from groundwood pulp and* Penicillium roqueforti *Thom when groundwood mats were inoculated as follows:

Upper left plate, control; upper right plate, *P. roqueforti* in central portion; lower left plate, *P. roqueforti* in central portion of mat containing bacterial isolate 5; and lower right plate, *P. roqueforti* in central portion of mat containing bacterial isolate 23.

1% suspension of groundwood pulp was sterilized so that duplicate mats could be made for each of the four following conditions:

A. Mats to be incubated as controls, i.e., neither bacteria nor mold were added.
B. Mats to be inoculated with *P. roqueforti* in the central portion.
C. Mats to be inoculated uniformly with isolate 5 prior to being made and finally inoculated in the central portion with *P. roqueforti*.
D. Mats to be inoculated uniformly with isolate 23 prior to being made and finally inoculated in the central portion with *P. roqueforti*.

The duplicate mats were incubated 14 days at 28°C. Figure 4 is a photograph of a set of four mats from this experiment. It is easy to see that isolates 5 and 23 definitely inhibited *P. roqueforti* on groundwood pulp by comparing the intensity of the dark areas which resulted from the mold growth. The mold grew much better on the mat in the upper right dish where no bacteria were added than on the mats in the two lower dishes where isolates 5 and 23 had been introduced.

The authors then proceeded to check isolates 5 and 23 for similarity and purity. Conventional methods of purifying bacterial cultures were used. It was soon found that isolate 5 contained both Gram-negative, nonspore-forming, short rods and Gram-positive, nonspore-forming, large rods. On the other hand, isolate 23 contained only Gram-negative, nonspore-forming, short rods which frequently occurred in pairs and which

grew well on nutrient agar with the production of greenish-yellow pigment. Both isolates 5 and 23 grew equally well on malt and nutrient agars; however, no greenish-yellow pigment was produced on malt agar. The color was white in this case. The authors checked purified subcultures of isolate 23 for antagonism toward *P. roqueforti*. In every case growth plates similar to Fig. 2 were obtained. Subcultures from isolate 5, on the other hand, exhibited much less antagonism and, in some cases, no antagonism was observed. Thus, it was shown that isolate 23 was a pure culture of a strongly antagonistic bacterium which effectively inhibited *P. roqueforti* both on malt agar and in groundwood pulp.

SUMMARY

In laboratory tests of a series of groundwood pulp specimens commercially treated with increasing amounts of phenylmercuric acetate, only the one containing the smallest amount of toxicant was free from mold. Since *P. roqueforti* was detected in the moldy batches of treated pulp and since this mold had already been shown to have a high resistance toward phenylmercuric acetate, it was suspected that biological competition between some microorganism producing a fungistatic substance and the mold might account for the anomaly of no mold growth at the smallest amount of treatment and heavy mold growth at significantly larger amounts of treatment. The authors successfully isolated a chromogenic, Gram-negative, nonspore-forming, short, rod-shaped bacterium which frequently occurred in pairs, and produced a strongly fungistatic substance. This type of biological competition was confirmed on malt agar and on groundwood pulp enriched with added nutrients.

As previously reported by the authors, limited growth of *P. roqueforti* apparently does not appreciably affect the usefulness of groundwood pulp. However, this mold organism seems to be widely distributed and the observed results thus add to the increasing amount of information available on biological competition between the members of the microflora of pulp and paper mills.

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Preparation and Testing of Mold-Resistant Wrapping Papers

LOUIS C. BARAIL

A great number of fungicides can be incorporated in pulp or paper. They are of various types: some are water soluble, some are only soluble in organic solvents, others are of the plastic type. The method of impregnation varies according to the chemical and physical characteristics. Some are more suitable for the incorporation in pulp, some others give best results when applied on the finished paper. The finished paper can be processed by dipping, soaking, spraying, painting, or laminating. In the second part of the paper the testing methods are described. It is necessary to test mildew resistant pulp and paper for performance against various types of fungi and other microorganisms, but other tests are also very important. For instance, the processing should not impart any undesirable odor or taste to the wrapper since such odor or taste would be readily transmitted to the goods wrapped in it. Such paper should not be toxic as it could transmit its toxic qualities to foods or other materials by contact. Continuous handling of treated wrappers with bare hands by employees in shipping departments should not cause the slightest skin irritation. Finally, the fungicides used in the treatment of wrappers should not change the normal color or texture of the paper, nor be susceptible to chemical changes due, for instance, to oxidation, to the action of light, cold or heat which would alter the normal color, feel or appearance of the wrapper.

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For many years the pulp and paper industry has been aware of the increasing damage caused to wrapping papers by microorganisms. The necessity of shipping goods of all kinds to all parts of the world resulted in conditions that were more favorable to their growth. For instance, shipping to foreign countries means more handling, changes in climatic conditions, and therefore necessitates far better protection. The increased periods of time during which perishable goods are stored, and also the various temperatures in which they are stored, require more severe specifications for wrappers of perishable goods. The packing in small containers of goods, that were formerly sold in bulk, almost immediately after they are manufactured, adds to the difficulties of long storage periods and has compelled the manufacturers of wrappers to investigate ways and means of protecting such paper containers and wrappers against destruction by bacteria and molds.

The second World War is responsible for important developments along these lines as many wrappers which had been used satisfactorily in the past were found to be of a rather poor quality when subjected to drastic wartime shipping conditions.

MILDEW PROOFING COMPOUNDS

We have investigated germicides and fungicides

that could be used for the protection of many kinds of materials. During these investigations, which lasted about six years, over 400 different formulae were tested not only for performance against a great many bacteria and fungi but also for their stability and harmlessness to human beings. It was very important to test fungicides at several temperature ranges to be sure that their properties would not be reduced or destroyed. There were also other considerations of a great importance such as the color and the toxicity of the products to be used. Quite a few products had to be eliminated for use in paper almost at the start because of their undesirable color or the objectionable odor they would impart to the pulp or the paper, or because of toxic and irritating properties they would transmit to foods, soaps, or cosmetics.

The various agents can be divided into three solubility classes:

1. Those that are soluble in water, or can be emulsified in water-soluble oils, or in emulsifiable compounds.

2. Those which are only soluble in organic solvents, the variety of which is a problem in itself.

3. The plastic-type compounds that can be applied only under certain conditions or cured at a high temperature.

Six ways of applying germicides and fungicides in the pulp and paper industry were found: one for incorporation into the pulp and five for application on the finished paper.

The application of fungicides into pulp is very well known in the pulp industry. It is basic impregnation and has definite specifications regarding solubility and resistance to temperature, the absence of odor and color, and the absence of primary irritants. The organic solvent type and the plastic type cannot be used in the pulp. We believe that the application on the finished paper is more rational, longer lasting, less expensive in the long run, and easier than the incorporation of an agent into pulp.

Finished papers can be processed five ways: dipping, soaking, spraying, painting, and laminating. Each of these methods has its advantages and specific uses.

The dipping process consists in passing the paper once or several times through a solution at a proper concentration of a germicide or a fungicide. This is a quick operation in which the moisture content of the paper is only slightly temporarily increased as the paper is dried immediately after the dipping process.

The soaking process is mostly used with fungicides which do not have an affinity for cellulose and which would be too expensive to use at a high concentration. Papers treated by the soaking process have to be dried at a rather high temperature without appreciable loss of their efficiency.

The spraying method is a very simple process by which all types of fungicides may be applied. The method is rapid, and the drying period that follows the spraying is very short. In the case of organic solvent fungicides it is not costly; however, it cannot be used with these volatile products or compounds whose fumes would be toxic to the operators.

The painting process or coating process consists in applying the fungicides by means of brushes or rollers. Such machines can process huge amounts of paper in a very short time, and the coating is more uniform than that obtained by spraying. Although it is sometimes used with water soluble compounds, it is definitely recommended for fungicides soluble in organic solvents or of the plastic type.

The laminating process is used when the consistency of the compound is an obstacle to its application to paper in the four other processes. This of course applies to the plastic type of compounds and to heavy materials which do not have any appreciable affinity for cellulosic materials.

The above processes have been used satisfactorily on various types of wrapping paper and cardboard which were efficiently made mildewproof and are used to wrap cake, soap and various foods. Wrappers of the glassine type are very efficient in preventing the growth of molds in bread, cakes, cookies, and pastry, for a substantial period of time. The fungicide used is of a volatile type and acts by the slow evaporation of its fumes. Other papers were found to be very satisfactory as a lining for cans and boxes containing tobacco, herbs, and similar products. Corrugated boxes are now treated with fungicides before being used for shipping foodstuffs such as cereals, dried fruits, and vegetables such as tomatoes, apples, and celery.

TEST METHODS

Mildew resistant pulp and papers are tested for efficiency by means of various methods. The first stage in the protection given by a treated wrapper consists in merely inhibiting the growth of molds without actually killing them. A wrapper with this property is fungistatic.

Fungistatic Tests

Although fungistatic tests give only a relative value of the efficiency of a fungicide, it is believed they are necessary and all compounds should always be tested for fungistatic properties. The results should always be interpreted from two angles. Because they are conducted in the same manner as the bacteriostatic tests (F. D. A. method), they only indicate the diffusion of the sample in the agar. Therefore, this diffusion should be studied in reference to both the diffusibility of the compound when incorporated in the material and the actual inhibitory or fungistatic value of the same compound. The samples are plated on Sabouraud maltose agar previously planted or inoculated with one or several fungi. As controls, samples of the untreated material are plated under the same conditions against the same fungus. The Petri dishes are examined after a reasonable period of time, and the minimum incubation time is that required to obtain a full growth of the culture. It will vary with the species and the temperature of incubation provided the medium remains the same.

Examination after this minimum period of time will give an indication of the agent's effectiveness, but it will not be sufficient. It very often happens that a compound diffuses quickly and widely on the surface of the agar so that within a few days a wide zone of inhibition is obtained. Then, a few days later, because of the diffusibility of the compound, the sample does not

contain a sufficient quantity to prevent a secondary growth inside the zone of inhibition. It is absolutely necessary to make a second observation of the plates from 5 to 8 days after the end of the minimum incubation period. The comparative study of the diffusibility and the actual fungistatic properties of wrappers as indicated by the width of the zone of inhibition and the presence or absence of a secondary zone of growth is of great value when perishable products are concerned, such as foods, cake soap, and bulk cosmetics.

Fungistatic and Fungus-Resistance Tests after Inoculation of the Material

These tests are more severe and of a greater informative value than simple fungistatic tests. They indicate not only whether or not a paper or paperboard will inhibit the growth of fungi but also whether or not the material, after direct inoculation, will sustain the mold as its food. The test consists of plating the samples in a Petri dish containing Sabouraud's agar which may or may not be planted with the test fungus, and inoculating the top of the material with a suspension of the same organism. At the end of an adequate incubation period, the plates are observed for inhibition, indicating fungistasis, and for the presence or absence of growth on top of the sample.

Fungicidal Tests

There are three ways of evaluating the fungicidal value of treated papers against fungi. The first two methods are empirical but simple, time saving, and correctly informative. The third method always gives reproducible and accurate results.

The first method consists in taking a loopful from the plate used in the fungistatic test as accurately as possible halfway between the edge of the sample and the edge of the inhibition zone. The loopful is then planted in another plate on Sabouraud's agar or in broth and incubated until growth appears in the control. The control is a loopful of an agar plate or slant of a fresh standard culture of the same organism and is plated under exactly the same conditions either on agar or in broth. Growth in the subculture from the fungistatic plate will indicate that the compound is fungistatic but not fungicidal. If no growth occurs in the subculture at the time that the control shows growth, it is wise to keep the sample in incubation for a few more days to be sure that the subculture is given all chances to grow under the best conditions.

Very often papers treated with a compound having a great diffusibility do not possess any fungicidal properties. The best results in this test are obtained with samples showing a medium size zone of inhibition and no secondary growth within it. On the other hand, it is obviously not advisable to make a fungicidal test on papers which show too limited an inhibition against fungi.

Fungicidal Tests after Inoculation of the Material. The procedure to determine the fungicidal value of a sample of material after inoculation consists of sampling from the top of the material at the end of the required incubation period when no growth is present on the sample. Sampling can be made with a dry or wet loop, or with wet sterile cotton, and the transfer is made either on a slant or in a dish on Sabouraud's agar. After the adequate period of inhibition, the tubes or plates are examined for the presence of growth.

This method is very simple and is satisfactory in a great number of cases. However, the contamination method gives more definite, rapid, and reproducible data on the fungicidal properties of compounds or treated papers when tested against cellulose-destroying fungi.

Fungicidal value after Contamination in Vitro. This test, which has been investigated in a great number of cases, is an adaptation to fungus testing of our method for the determination of the bactericidal value after contamination. The method, developed a few years ago and published at that time, has been tried and adopted by several bacteriological laboratories for the testing of germicides. Recent experiments have definitely proved that it can be satisfactorily adapted to the testing of fungicides, whether the test organisms are pathogenic or not.

The purpose of the method is to determine whether materials treated with fungicides have fungicidal properties and retain these fungicidal properties for various periods of time after a certain number of washings or leachings, after being immersed or soaked in water or other liquids, or after being used for packing goods having a relatively high moisture content.

The test is very severe since all materials are contaminated with a fresh test culture of a fungus, or a mixed culture of fungi, by total immersion before being submitted to the tests. We generally use in tests on papers *Chaetomium globosum* or *Aspergillus niger*, but any fungus, pathogenic or not, can be used.

The test is conducted as follows: Three disks, $^5/_8$ inch in diameter, are cut from each sample of paper treated with the fungicide being tested. Three test disks are also taken after each washing, leaching, or soaking or after five washing, leaching, or soaking-time units. Each group is placed successively in a tube containing 5 ml. of a fresh suspension of a standard test fungus for 1 minute. The suspension can be made in any sterile solution, such as distilled water, saline, or broth, as tests have proved that the suspension medium does not have any influence on the results of the tests. Upon removal, the first disk is placed with aseptic precautions in a tube of sterile nutrient broth and swirled for 5 minutes, removed to a second tube of sterile nutrient broth and swirled for 10 seconds, and finally transferred to a third tube of sterile broth. A second disk is transferred to a first tube of broth and swirled for 10 minutes, and then to a second tube of broth and swirled for 10 seconds, and then removed to a third tube. The third disk is transferred to a first tube of broth and swirled for 15 minutes, and then to a second tube of broth and swirled for 10 seconds, and finally removed to a third tube. As controls, three disks cut from the untreated material are handled the same way as the three treated disks. All 18 tubes are incubated in a high humidity chamber at a temperature which can vary from 24 to 37°C. according to the normal conditions of use of the paper being tested. Sabouraud's agar Petri dishes can be used instead of broth tubes if found more convenient. The results are the same in both cases. The incubation lasts until there is a copious growth in the control tubes. At that time, the test tubes are examined for the presence of growth.

This method is applicable to all kinds of fungicides whether readily soluble in water and having a quick diffusion or rather insoluble in water and having a high power of penetration in the material. The method is also applicable to laminated fungicides of the plastic type. In this case, it is sometimes necessary to use sterile oil or solvents to conduct the tests. It is of course mandatory to use solvents which do not have any fungicidal value *per se*. Results for the first group of fungicides are more specific in the first transfer tube. Results of the second transfer tube, where the excess of fungicide is washed out, have only an informative and not conclusive value. Finally, this method can be advantageously used to replace the old phenol coefficient method which is not applicable to fungi.

TESTS FOR HARMLESSNESS

In order to find out whether mildew resistant wrappers can be used for wrapping foodstuffs, it is necessary to conduct toxicity and other tests.

Toxicity Tests

The evaluation of the toxicity of a fungicide or of a paper treated with a fungicide is determined by the minimum lethal dose. Various amounts of several dilutions of the compound or various amounts of extracts of the treated papers are injected into mice to determine the smallest amount of chemical that will cause the death of all injected animals, or of half of them. The results are generally expressed in grams or milligrams by kilogram of body weight or by the minimum dose that would kill a man weighing 70 kg., (154 pounds). Such tests have shown very great differences in toxicity between the most efficient fungicides.

The determination of the minimum lethal dose is often replaced by feeding tests conducted on animals for periods of time which can vary from one week to their life time. The animals are fed a mixture of their normal diet, and amounts of the fungicide that can vary from 5 to 20% of their diet. The Food and Drug Administration very often requires that such tests be conducted for a long duration in order to insure complete protection of the public when these wrappers are used in contact with food.

Skin Irritation Tests

The presence of a primary irritating substance in mildew-resistant papers is very important when such wrappers are handled all day long in packing and shipping departments. A great many fungicides are known to cause irritation of the skin either primarily or after having previously sensitized the skin. It is, therefore, advisable to conduct these tests on the fungicides first on animals, and later on, on human beings at the concentrations of use in the wrappers.

Taste Tests

In order to be sure that no disagreeable taste will be imparted to foods by the wrappers, taste tests are always conducted, and panels of tasters as large as possible are used.

Odor Measurement Tests

The detection of objectionable odors which can be transmitted by the wrapper to such products as foods, soaps, and cosmetics is made with great precision by means of the Osmometer. The Osmometer is the first apparatus wherein the elimination of variables has been so perfected that the testing of an odor can be made by several individuals in good health and still bring result with variations of less than 5%. This shows that by elimination of all variables the organoleptic errors are reduced to a minimum. The Osmometer is based on the air-dilution principle using quantities of odor-free and odorous air which are diluted and mixed in various concentrations. The dilution is performed under moderate pressure which can be made to vary from one to 300 mm. Hg. However, pressures higher than 150 mm. are seldom used.

If no odor is detected when the air is suddenly released from the mixing flask the experiment is repeated, this time with twice the volume of air forced through the specimen. Other readings are made until sufficient air has been pumped through the odorous object to produce the sensation of odor. The concentration at which the odor becomes just barely perceptible is called the "threshold value."

This threshold value can be expressed numerically and this numerical expression is called the "odor threshold number." The ratio between the total final pressure and the pressure increment caused by the odorous air results in the odor threshold number:

$$\text{Odor threshold number} = \frac{\text{Total pressure}}{\text{Odorous increment}}$$

The odor threshold number gives the concentration of the odor in the air dilution which will just barely cause the sensation of odor. For strongly odorous substances, this concentration as reported in the odor threshold number will be low, and the odor threshold number will be high. For weak odors it will be the opposite.

The following classification of odor threshold numbers is used for the evaluation of the results of the tests. A threshold number in the range from 1.0 to 5.9 means a very slight odor; from 6.0 to 8.3: slight odor; from 8.4 to 13.0: definite or objectionable odor; from 13.1 to 26.0: strong odor; from 26.1 to 91.0: extremely strong odor.

CONCLUSIONS

Out of more than four hundred compounds which were tested for comparative germicidal and fungicidal value, very few were found to meet all the requirements for efficiency: lacking of odor; color, and other changes; and toxicity.

Chlorine derivatives are very efficient when incorporated into the pulp. When there is no objection to odor, pentachlorphenol and sodium pentachlorphenate can also be recommended. When there is no objection to color, copper compounds give satisfactory results. However, these two groups of chemicals are relatively toxic. The best results are definitely obtained with long-chain mercurials which besides being nontoxic and nonirritating at normal concentrations of use, have lasting qualities which no other compounds do possess.

The pulp and paper industry now has at its disposal a variety of products which can solve most of its problems dealing with bacteria and mold contamination.

Received March 15, 1950. Presented at the Annual Meeting of the Technical Association of the Pulp & Paper Industry, New York, N. Y., Feb. 20–23, 1950.

Fundamental Research Conference

The recent Fundamental Research Conference on the subject of "The Effect of Fiber Characteristics on Paper and Papermaking Properties," sponsored jointly by the Fundamental Research Committees of the Technical Section of the Canadian Pulp and Paper Association and of TAPPI, maintained the quality of program and high level of discussion apparent in the earlier fundamental research conferences held in Detroit.

Sessions were held on Monday, Tuesday, and Wednesday, May 29, 30, and 31, using the facilities of the Chateau Frontenac at Quebec, P. Q. The interest of those participating in the conference was such that meetings could begin promptly in the morning, and there seemed to be no falling off in attendance throughout the long discussions of the day. The program in its final form follows.

A. The Individual Fiber Properties Related to Papermaking

1. *Fiber Dimensions and Physical Structure*

Discussion Leader—C. B. Purves, McGill University, Montreal, P. Q.

(a) "The Effect of Variations in Size and Shape of the Fibers," by Gerald Haywood, West Virginia Pulp & Paper Co., Luke, Md.

(b) "The Orientation of the Cellulose in Wood Fibers," by George L. Clark, University of Illinois, Urbana, Ill.

(c) "The Proportion of Water-Accessible and Non-accessible Cellulose in Wood Pulps," by Merle Heath, The Institute of Paper Chemistry, Appleton, Wis.

(d) "The Role of Pulp Viscosity and the Degree of Polymerization of Cellulose on the Papermaking Properties," by M. O. Schur, Ecusta Paper Corp., Pisgah Forest, N. C. and Harry F. Lewis, The Institute of Paper Chemistry, Appleton, Wis.

2. *Strength and Mechanical Properties*

Discussion Leader—Milton Harris, Milton Harris Associates, Washington, D. C.

(a) A Discussion of Some of the Problems to be Met in Developing an Analytical Approach to the Study of the Elastic and Rheological Properties of Papermaking Fibers," by J. A. Van den Akker, The Institute of Paper Chemistry.

(b) "Fundamental Research in the Textile Industry. The Relation Between the Chemical Constitution of Protein Fibers and Their Use Requirements," by Milton Harris, Milton Harris Associates.

3. *Surface Properties and Measurements*

Discussion Leader—J. d'A. Clark, Consulting Engineer, Longview, Wash.

(a) "The Specific Surface of Fibers, Measurement and Application," by S. G. Mason, Pulp and Paper Research Institute of Canada, Montreal, P. Q.

(b) "Notes on the Determination of the Specific Surface of Pulps by the Silvering Method," by B. L. Browning, The Institute of Paper Chemistry.

(c) "Electrokinetic Characteristics of Fibers," by S. G. Mason, Pulp & Paper Research Institute of Canada.

(d) "Fiber Surface Effects of Fibers in Their Relation to Papermaking," by Harry F. Lewis, The Institute of Paper Chemistry.

B. Inter-Fiber Properties Related to Papermaking

1. *Fiber-Water Relationships*

Discussion Leader—W. Boyd Campbell, Pulp and Paper Research Institute of Canada, Montreal, P. Q.

(a) "General Principles of Polymer Swelling," by Herman Mark, Polytechnic Institute of Brooklyn.

(b) "Swelling of Cellulose Fibers, Its Measurement and Significance," by W. Gallay, The E. B. Eddy Co., Hull, P. Q.

(c) "Effect of Drying and Heating on the Swelling and Strength of Fibers," by W. Galay, The E. B. Eddy Co.

(d) "Bound Water and Hydration," by Alfred J. Stamm, U. S. Forest Products Laboratory, Madison, Wis.

(e) "Flocculation and Formation," by S. G. Mason, Pulp and Paper Research Institute of Canada.

2. *Fiber-Fiber Bonding*

Discussion Leader—C. C. Heritage, Wood Conversion Co., St. Paul, Minn.

(a) "The Measurement and Nature of Fiber-Fiber Bond," by Loren V. Forman, The Institute of Paper Chemistry.

(b) "Effects of Natural Beater Additives on Papermaking Fibers," by John Swanson, The Institute of Paper Chemistry.

(c) "Progress and Problems in the Bonding of Fibers by Synthetic Resins," by Chester Landes, American Cyanamid Corp., Stanford, Conn.

(d) "Preliminary Results on the Nature of the Paper Bond by Studies of Softening with Amides," by Börje Steenberg, Swedish Forest Products Research Institute, Stockholm, Sweden.

C. Chemical Composition of Fibers in Relation to Papermaking Properties

Discussion Leader—Harry F. Lewis, The Institute of Paper Chemistry.

1. The Influence of Hemicelluloses in Wood Pulp Fibers on Their Papermaking Properties," by L. G. Cottrall, Albert E. Reed and Co., Ltd., England.

No attempt will be made to abstract the various papers at this time, for arrangements have been made to carry the papers and the complete discussions in the August and September issues of *Tappi* and also of the Canadian magazine. The discussions were spirited, and it is unfortunate that every member of TAPPI could not have been present to hear them. They continued on into the night over the tables in the French restaurants of Quebec and up and down the boardwalk overlooking the St. Lawrence River.

One of the high points of the meeting was the observation by Dr. Stamm that a new chemical method developed at the Forest Products Laboratory for determining the relative amounts of accessible and nonaccessible cellulose checked very well with the physical method described by Dr. Heath involving the accessibility of pulps to water—a purely physical method.

Dr. Milton Harris gave a masterly presentation of the place of fundamental research in the textile industry, with particular reference to the chemical structure of protein fibers and their use requirements, showing his listeners how much there was alike between the problems of the textile and the paper industries.

Dr. Mason's paper on the specific surface of fibers in the third session precipitated an extremely active discussion between proponents of the method for determining specific surface by silvering, by optical methods, and by permeability to water.

On the afternoon of the second day Professor Mark of the Polytechnic Institute of Brooklyn gave a fine review of the general principles of polymer swelling. This paper proved to be extremely stimulating and furnished the background of a great deal of discussion during the afternoon session which was high lighted by the attempts of the members of the audience

to reach a common agreement as to the significance of swelling of cellulose fibers and the mechanism of flocculation and formation.

The discussion leader of the fifth session was C. C. Heritage, who was President of TAPPI when the Fundamental Research Committee was organized. As President, he asked Dr. Harry F. Lewis, the present Chairman of the TAPPI Committee to accept that responsibility. The paper presented by Loren Forman of The Institute of Paper Chemistry on "The Measurement and Nature of Fiber-to-Fiber Bonding" in itself justified the earlier enthusiasm of the then President of TAPPI for the development of fundamental information.

Mr. Cottrall's paper on the "Effect of Hemicelluloses on the Papermaking Properties of Pulps" likewise proved to be stimulating to the group and provoked a large amount of discussion.

In addition to being international in the sense that the meeting was sponsored by both the Canadian and U. S. Technical Associations, it was international in the sense that in the audience and among the speakers were a number of representatives of foreign countries, including in the group Mr. L. G. Cottrall of Albert E. Reed & Co., Ltd. of England representing the British Paper and Board Association, Dr. Börje Steenberg of the Swedish Forest Products Research Institute who gave a short review of some preliminary work on the nature of the paper bond by studies of softening with amides, as an added paper to the program. The Canadian Association proved to be a very fine host, and good spirit prevailed throughout. For many, the meeting provided the first opportunity to visit the quaint, old city of Quebec, and advantage was taken of the spare time on Sunday and early evening to go around the city in the horse-drawn carriages and on foot; after experience with the taxi cab drivers on arrival, few of the visiting TAPPI members cared to risk their life and limbs seeing the city by taxi.

TAPPI Testing Conference

The Testing Conference will initiate the series of fall meetings of the Technical Association of the Pulp and Paper Industry. It will be held at the plant of the Eastman Kodak Co., Kodak Park, Rochester, N. Y., on Sept. 13–15, 1950.

The Seneca Hotel, which is located near to Kodak Park, has been designated as headquarters for housing purposes. Reservations should be sent, as soon as possible to Mr. Earl F. Campbell, Resident Manager, who should be advised that the reservation is for the TAPPI Testing Conference. Mr. Campbell has requested that as many as possible share twin-bedded double rooms. Although the Seneca, with 500 rooms is the largest in Rochester, its midweek capacity is usually filled to the limit. A limited number of single rooms will be available. Rooms will be available on the evening of Tuesday, September 12 for those who wish them. An advance deposit will assure possession of rooms upon arrival.

This will be the second Testing Conference. The first was held in October, 1948 at the Mellon Institute in Pittsburgh, Pa. About 175 attended and a considerable amount of effective work was accomplished.

The Testing Conferences are essentially work meetings and differ in nature from the usual fall meetings of the Association in that no formal papers are presented. The preparation of the TAPPI Standards has been one of the great contributions of the Technical Association to the progress of the industry. Over the years a large number of standard testing procedures have been developed. This program proceeded rather rapidly, but as the problems became more complex the need for conferences such as these became apparent. The Pittsburgh Conference broke the log-jam of complications since it brought together individuals and data that made it possible to agree on many testing details.

Meetings such as the one proposed for Rochester will need to be held in the future to assure steady progress since the job of preparing standards is becoming more difficult and much work needs to be done in the intervals between these conferences to acquire test data that will demonstrate the reliability and reproducibility of new methods.

Just as the Association was indebted to the Mellon Institute in 1948 for permitting the Testing Division to use its exceptionally fine meeting facilities and providing a guided tour of its unusual laboratories, the Division, this fall, will be indebted to the hospitality of the Eastman Kodak Co. for providing meeting rooms and a visit to some of its operating departments.

James d'A. Clark, Research and Development Engineer, Longview, Wash., is General Chairman of the Testing Division and has been an aggressive leader in the field of testing procedures. Dr. Clark, while serving as a Lt. Col. in the Quartermaster Corps. during the recent war added to his already high prestige in the work that he did in developing the then famous CQD packaging specifications. His industry in connection with the establishment of TAPPI Standards is well known.

Although it is not yet possible to indicate the time schedule for the separate committee-sponsored sessions at Rochester the following paragraphs will outline present plans regarding the programs. As in the case of the Pittsburgh meeting the program at Rochester will tend to follow the interests of those present.

Optical Properties Committee

A. H. Croup of the Hammermill Paper Co., Erie, Pa., and Chairman of the Optical Properties Committee advises that his committee will discuss the following:

1. The final draft of the proposed tentative standard for gloss determination.
2. The results of the use of the National Bureau of Standards fading papers for calibrating fadeometers and the possibility of recommending these for general use in the industry.

Chemical Methods Committee

Milton Fillius, Eastman Kodak Co., Rochester, N. Y., and Chairman of the Chemical Methods Committee has indicated that his committee will sponsor two 3-hour sessions.

Session 1.
A. The distillation step in the method for the determination of pentosans in pulp and wood.
B. Methods for the moisture determination of pulp.
C. Alternate subject—A rapid method for the determination of nitrogen.

Session 2.
A. The bleach consumption of pulp. A discussion of three TAPPI methods: T 214, Permanganate Number of Pulp; T 219, Bleach Requirements of Pulp; and T 202, Chlorine Consumption of Pulp.
B. The chemical analysis of papers containing wet-strength resins. A discussion of the complications introduced into the analysis of paper for alpha cellulose, copper number, etc., by the presence of wet-strength resins.

Mr. Fillius has requested that any member who has had experience with these methods write him giving suggestions, based on substantiating data, if possible, that will aid the committee in improving the techniques involved in these methods.

Packaging Materials Testing Committee

L. E. Simerl, Marathon Corp., Menasha, Wis., and Chairman of the Packaging Materials Testing Committee has indicated that the following subjects will be discussed in two 3-hour sessions.

1. Project 277 Water Vapor Permeability at Zero F.
2. Project 328 Flavor and Odor Transfer.

LOOK AT ALL THAT GOOD STOCK

all purchased and processed — all ready to go onto the paper machine and be made into good, salable paper — all rescued from the river by

BIRD SAVE-ALLS

Don't let those expensive fibres go to waste. Under today's conditions Bird Save-Alls pay for themselves in no time. May we supply recommendations, layouts and estimates?

BIRD MACHINE COMPANY
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Mass.

3. Project 330 Testing of Pressure Sensitive Tapes.
4. Project 357 Water Vapor Permeability of Completed Packages.
5. Project 275 Flexibility at Low Temperatures (including coating materials, tapes, laminated products, etc.).
6. Project 331 Heat Sealability.
7. Review of Assigned Tests (T 446, 448, 463, 464, 465, 473, 474, 475, 477, and 806).
8. New Projects.

Fibrous Materials Testing Committee

E. R. Schafer, Forest Products Laboratory, Madison, Wis., and Chairman of the Fibrous Materials Committee is not planning to hold a session at the Rochester meeting. However, he has indicated that B. F. Kukachka of the Laboratory will present a paper at the general session on "A Universal System for Wood Identification." By the time the conference is held Dr. Kukachka will have returned from the meeting of the International Association of Wood Anatomists to be held in Stockholm, Sweden, where some of the problems involved in the development of an international code will have been discussed.

Paper Testing Committee

One 3-hour session has been reserved for the Paper Testing Committee. A. K. Warner, Skinner & Sherman, Inc., Boston, Mass., and Chairman of the Paper Testing Committee has not yet decided upon the complete program but it will include a discussion of and possible recommendation to adopt a newly standardized procedure for the bursting test which has been developed mainly by the corresponding Canadian committee.

Nonfibrous Materials Testing Committee

The Nonfibrous Materials Testing Committee, James P. Casey, New York State College of Forestry, Syracuse, N. Y., Chairman, will hold two and possibly three sessions at the Rochester Conference. The major part of the program will be devoted to (1) Testing of Starches, (2) Analysis of Mineral Fillers, (3) Analysis of Rosin Size, and (4) Testing of Clays. Other methods that may receive preliminary consideration are Analysis of Wax-Rosin Size, Testing of Sodium Aluminate, and Testing of Defoaming Agents. If time permits the following existing methods will be reviewed: Gasoline T 601, Coal T 605, Analytical Reagents T 606, Casein T 607, Indicators T 609, Standard Volumetric Solutions T 610, and Sodium Silicate T 632. Any members who may wish to comment on any of these are urged to write to Mr. Casey.

Microscopy Committee

C. E. Brandon, Aetna Div., Howard Paper Mills, Inc., Dayton, Ohio, and Chairman of the Microscopy Committee advises that his committee will sponsor two 3-hour sessions.

Session 1.
Discussion of the proposed revision of T 401-42 (Fiber Composition of Paper).
(a) The differentiation of alpha-treated softwood sulphite and highly bleached sulphite with the C-stain.
(b) The effect of curlating on the C-stain.
(c) A new stain for the differentiation of bleached sulphite and bleached sulphate.
(d) General discussion of T 401 m-42 and of any new stains.

Session 2
1. Discussion of the revision of T-8 (Species Identification of Wood and Wood Fibers).
2. The use of the microscope in analyzing for mineral fillers in papers.
3. Test for elemental sulphur in pulp and paper.

A joint meeting will be be held with the Pulp Testing Committee to discuss:

1. The measurement of fiber length
   (a) Optical method of the Eastman Kodak Co. Laboratory.
   (b) Routine method of the Alton Box Board Co. Laboratory.
2. Discussion of a method for "The Determination of the Amount of Dirt in Pulp (Revision of T 213 m-43)."

Pulp Testing Committee

L. V. Forman, Institute of Paper Chemistry, Appleton, Wis., and Chairman of the Pulp Testing Committee advises that one session will be sponsored by his committee. Another session will be held jointly with the Microscopy Committee as already described. Other projects of the Committee that will receive attention are (1) Zero-span Breaking Length of Pulp and (2) Pebble Mill Processing of Pulp, and (3) Dirt in Pulp.

Wax Testing Committee

As in the case of the Pittsburgh Testing Conference the Wax Testing Committee, A. M. Heald, Marathon Corp., Menasha, Wis., Chairman, will have the largest attendance and hold the most extensive program at the Rochester Conference. This committee which works closely with the ASTM as a TAPPI-ASTM Joint Committee on Petroleum Wax will sponsor four 3-hour sessions.

Although Dr. Heald has not yet announced his program it is expected that there will be discussions on the reports of the following subcommittees: (1) Strength Tests; (2) Flow Tests; (3) Stability Tests; (4) Sensory Tests; (5) Composition (Melting Point and Oil Content, Melting Point Distribution); (6) Blocking; (7) Sealing and Laminating; and (8) Scuff-Gloss.

Container Testing Committee

The Container Testing Committee, W. J. Balster, Don L. Quinn Laboratory, Chicago, Ill., has not yet announced its agenda.

Graphic Arts Committee

Although the Graphic Arts Committee is not included in the organization of the Testing Division the Testing Conference is open to any committee in the Association that deals with testing methods. Fred D. Elliott, Time and Life Laboratories, Springdale, Conn., Chairman, has advised that one 3-hour session will be devoted to an open round-table discussion on printability to establish the properties that are important and should be tested by the papermaker. The following will be considered:

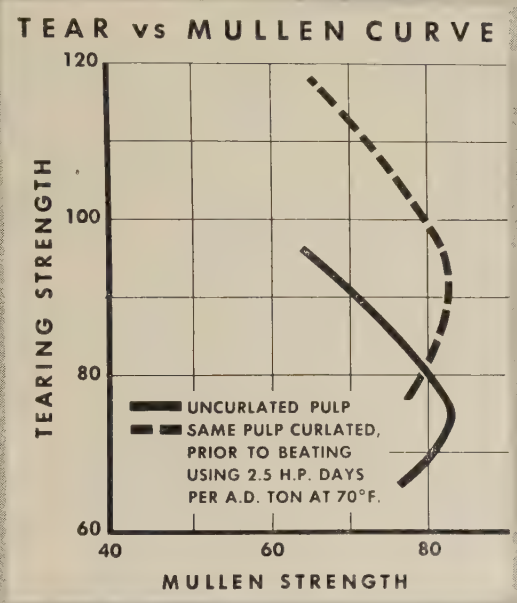
(*a*) A brief report on the major grades of papers, their principal end uses, and percentage of the various grades printed by each of the three printing processes.
(*b*) Discussion of the important physical characteristics needed by a given sheet to perform and print properly when run by the letterpress, gravure, and offset processes. (The purpose of this discussion is to obtain an opinion on what the papermaker should be testing in a sheet being manufactured for a specific type of press or method of printing).
(*c*) Review and discussion of various existing tests pointed toward studying their suitability for determining characteristics considered important in (b).
(*d*) Discussion of new and not generally accepted tests such as the Brush surface analyzer for surface smoothness determinations.
(*e*) Discussion of means of checking certain important characteristics for which there is no present adequate test such as the effect of capillarity or pinholes in paper on ink penetration or how to determine the water solubility of coated offset paper.

General

The purpose of the Testing Conference is to develop new testing methods and to improve those now established as standards. Any member of the Technical Association or related societies is welcome to attend, particularly if he is qualified to contribute to the discussions mentioned. Each of the committee chairmen will welcome advice relative to these methods and will be glad to receive suggested outlines of new methods. The discussion will be expedited if members planning to attend will write to any of the committee chairmen about any testing method falling within the scope of his committee.

Curlators Are Proving Their Value

Effect of Curlation prior to beating on the tear-mullen relationship during beating of a previously undried northern unbleached sulphite pulp.



TEST DATA

	HP/DT*	FREENESS	MULLEN	TEAR	DENSO-METER
UNCURLATED PULP	0	840	66.8	93	13
	1	810	77.6	83	34
	2	764	80.8	77	95
	3	672	84.0	75	260
	4	615	81.5	74	580
	5	521	70.8	60	1330
	7	394	66.5	55	3750
	9	285	64.6	54	5660
PREVIOUSLY CURLATED PULP					
	0	836	66.2	117	3
	1	770	82.8	95	22
	2	688	80.2	81	125
	3	575	76.8	72	360
	5	390	71.7	67	2640
	7	270	55.0	48	8280

*H. P. DAYS PER A. D. TON SPENT IN BEATING ONLY.

Note: Tear at maximum mullen of uncurlated pulp is 75, of curlated pulp 95.

Description of testing procedures employed by the Curlator Corporation Laboratory will be provided upon request.

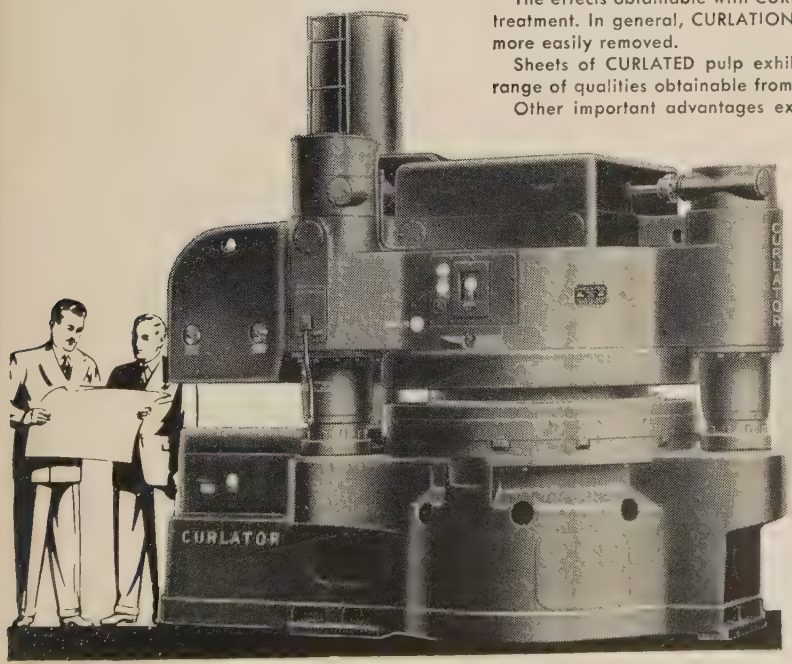
CURLATION is a new mechanical process for improving pulp properties, fully tested and thoroughly proved. Curlation not only produces a permanent change in the shape of pulp fibres, but also exerts a powerful de-shiving action, with negligible change in freeness.

The effects obtainable with CURLATION vary widely with the raw material and the conditions of treatment. In general, CURLATION tends to produce an easier beating pulp from which water is more easily removed.

Sheets of CURLATED pulp exhibit a more even textured matte appearance. In addition, the range of qualities obtainable from a given pulp is broadened.

Other important advantages exist . . . WRITE for new bulletin C-2 on the C-50 CURLATOR.

The CURLATOR is a tool for altering the properties of pulp and paper to obtain qualities hitherto unavailable by mechanical means. The effects of CURLATION vary with the raw material and may be described only in relation to a particular pulp.



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It is possible that there will also be a meeting of the Structural Fibrous Materials Committee to discuss tests for structural boards.

Fifth TAPPI Engineering Conference

The Fifth Engineering Conference of the Pulp and Paper Industry, sponsored by the Engineering Division of the Technical Association of the Pulp and Paper Industry, will be held at the Netherland Plaza Hotel, Cincinnati, Ohio, on Oct. 2–5, 1950. George H. Pringle, Asst. Chief Engineer of the Mead Corp., will preside as General Chairman.

Following is the preliminary program:

Mill Design and Economic Aspects Session

ALVIN H. JOHNSON, Consulting Engineer, New York, N. Y., *Chairman.*

"Paper Machine Drives," by Roland A. Packard, Holyoke Machine Co., Holyoke, Mass. (This paper will be a continuation and further development of Mr. Packard's paper given at the Fourth Engineering Conference at Boston.)

M. C. Celli of the Rust Engineering Co., Pittsburgh, Pa., will present another paper on mill construction, supplementing his paper given at Boston.

The National Council for Steam Improvement is preparing a paper dealing with some of the engineering aspects of handling mill wastes and white water.

Materials Handling Session

A. P. SCHNYDER, Ebasco Services, Inc., New York, N. Y., *Chairman.*

"Outstanding Practices in Materials Handling at Pacific Coast Mills," by F. D. Helverson, Crown Zellerbach Corp., Portland, Ore.

"Materials Handling of Finished Pulp and Paper from Machine to Shipping Dock," by H. F. R. Weber, Link-Belt Co., Chicago, Ill.

Mill Maintenance and Materials Session

H. F. PARKER, New York & Pennsylvania Co., Lock Haven, Pa., *Chairman.*

Teflon in Process Equipment," by D. B. Hanson, E. I. du Pont de Nemours & Co., Inc., Wilmington, Del.

"Slotted Screen Plates for Pulp and Paper Mills," by Henry W. Downs, Jr., Union Screen Plate Co. of Canada, Ltd., Lennoxville, P. Q.

"Maintenance of Roofs," by G. F. Hrubecky, Marathon Corp., Menasha, Wis.

Engineering Research and Machine Design Session

P. H. GOLDSMITH, Pusey & Jones Corp., Wilmington, Del., *Chairman.*

"Gear Unit and Gear Motor Application in the Pulp and Paper Industry," by F. S. McBride, Westinghouse Electric Corp., E. Pittsburgh, Pa.

"Air Removal from Stock Ahead of the Head Box," by C. L. Clark, Clark & Vicario, Bronxville, N. Y.

"Thoughts on Standardization," by M. Click, Sandy Hill Iron & Brass Works, Hudson Falls, N. Y.

Electrical Engineering Session

ALBERT S. GOODRICH, Hammermill Paper Co., Erie, Pa., *Chairman.*

"The Selection of Electric Motors for Paper Mill Application," by R. F. Sorenson, Union Bag & Paper Corp., Savannah, Ga.

A discussion period will be devoted to a consideration of the committee report on "Couch Power Requirements."

Chemical Engineering Session

J. R. LIENTZ, Union Bag & Paper Corp., Savannah, Ga., *Chairman.*

The Kraft Digester Corrosion Subcommittee will report at this session. M. A. Schiel, A. O. Smith Corp., Milwaukee, Wis., will discuss some aspects of digester construction.

Steam and Power Session

C. J. SIBLER, West Virginia Pulp & Paper Co., New York, N. Y., *Chairman.*

"Installation and Operation of the 1200-lb. Steam Plant at the Union Bag & Paper Corp. Mill," by H. R. Arnold, Union Bag & Paper Corp., Savannah, Ga.

"Steam Plant Modernization of a Medium Size Mill," by A. E. Crane.

"The Dual Circulation Boiler," by P. W. Foster, Foster-Wheeler Corp., New York, N. Y.

Drying and Ventilating Session

M. L. BARKER, Beckett Paper Co., Hamilton, Ohio, *Chairman.*

"Drying 800-Tons Kraft Paper Per Day on Our Machine," by L. C. Crowder, Macon Kraft Co., Macon, Ga.

Hydraulics Session

K. J. MACKENZIE, Eastman Kodak Co., Rochester, N. Y., *Chairman.*

A discussion period will be devoted to further consideration of the Committee Project "Flow of Paper Stock in Pipes."

Data Sheets Session

HENRY J. PERRY, Paper Trade Journal, New York, N. Y. *Chairman.*

Mr. Perry will present a progress report dealing with the current activities of the committee.

Other Features of the Conference

The Ohio Section and a special committee under the chairmanship of Tany Agronin, Shartle Bros. Machine Co., Middletown, Ohio, is preparing an interesting program of plant visits, banquet, etc., which will add to the enjoyment of the Conference.

Room reservations at the Netherland-Plaza Hotel should be made direct. The Technical Association will soon send out a mailing which will include a reservation card. Individuals making direct reservations should address their requests to William Pfeiffer, Sales Manager of the Hotel, mentioning the Fifth Engineering Conference of the Pulp and Paper Industry. Located near the Netherland-Plaza Hotel and under the same management is the Terrace Plaza Hotel. This hotel has received considerable general publicity because of its ultramodern room facilities. It will be a very desirable place to reside during the Conference.

TAPPI Plastics Conference

The TAPPI Plastics Conference will be held at the New York State College of Forestry. Syracuse, N. Y., on Oct. 19–20, 1950. Hotel accommodations will be available at the Hotel Onondaga and the Hotel Syracuse. E. C. Jahn of the New York State College of Forestry is Chairman of the TAPPI Plastics Committee sponsoring the conference.

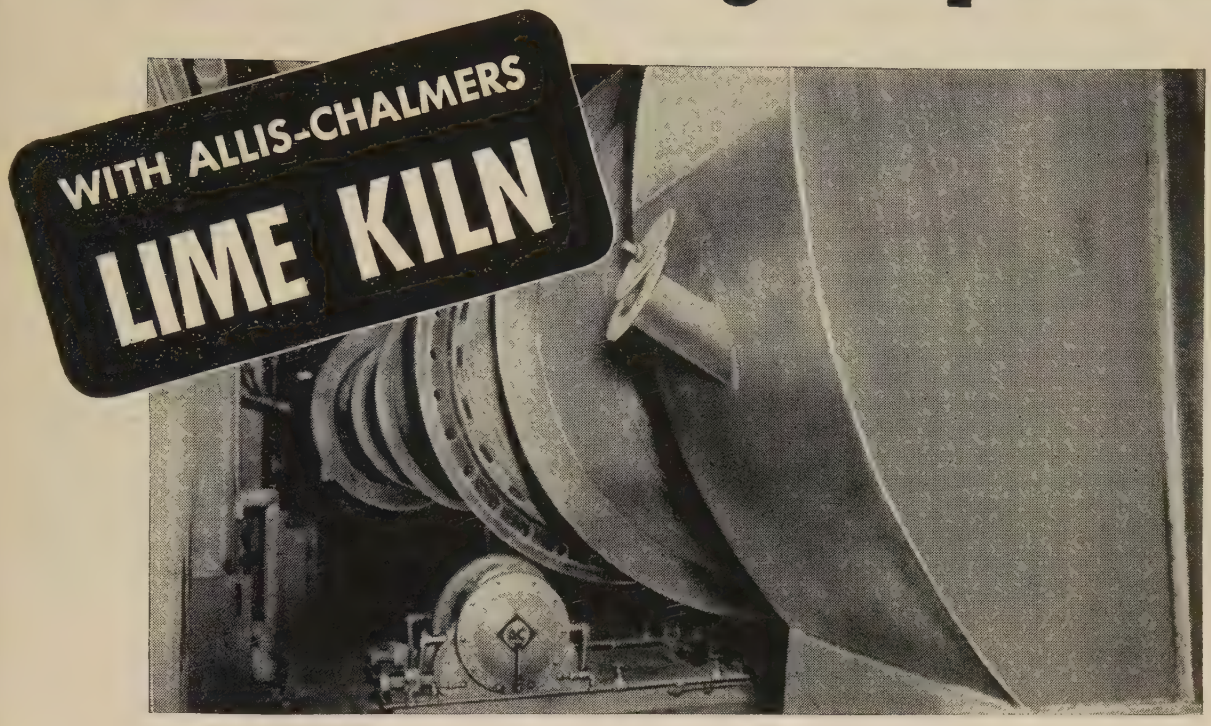
The themes of the four sessions will be as follows:

1. Properties of Plastics in Relation to Papermaking.
2. Beater Addition Fundamentals.
3. Fundamental Properties of Cellulose in Relation to Plastics.
4. Coating Fundamentals.

Fibrous Agricultural Residues Conference

The TAPPI Fibrous Agricultural Residues Conference will be held at the Northern Regional Research Laboratory, Peoria, Ill., on Nov. 12–13, 1950. Samuel I. Aronovsky, Chairman, advises that a block of rooms have been reserved at the Pere Marquette Hotel for these dates. A dinner will be held at Pabst Hotel on the evening of November 13. Details concerning the technical program will be published in future issues of *Tappi.*

More Heating Surface; Fuel Economy Improved!



A-C KILN GIVES CROWN-ZELLERBACH KRAFT MILL THESE BENEFITS:

- Heat recuperating chain system saves fuel.
- Kiln design provides continuity of operation.
- Maximum capacity at lowest possible fuel consumption.

INCREASED KILN CAPACITY was obtained at the Camas, Washington, kraft mill of Crown-Zellerbach corporation with the installation of a new 8 x 120 ft rotary type Allis-Chalmers lime kiln.

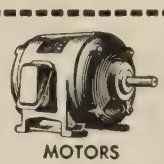
Feed end of kiln is equipped with a heat recuperating chain system which efficiently transfers heat from hot gases to wet material. This chain system provides a large heating surface which effects the exchange of heat, gives low flue gas temperature, cuts dust loss.

You can get similar efficiency in your lime sludge recovery with Allis-Chalmers kilns. Design features include: ▸ floating type steel riding rings; ▸ steel rollers; ▸ heat recuperating chain system. Allis-Chalmers kilns are designed to eliminate effects of torsional vibration.

Allis-Chalmers also supplies feeders, slakers, burning equipment, dust collectors and fans, and master control panel equipment. Teamwork of A-C kilns with Allis-Chalmers motors and Texrope V-belt drives provides one manufacturer responsibility for successful operation.

For more details, see your nearby Allis-Chalmers sales office or write direct for Bulletin 07B6368. A-2872

ALLIS-CHALMERS, 828A SO. 70 ST.
MILWAUKEE, WIS.

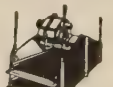

MOTORS

CENTRIFUGAL PUMPS

TEXROPE V-BELT DRIVES

STREAMBARKER

HI-DENSITY FEEDER


VIBRATING SCREENS

AND OTHER EQUIPMENT FOR PAPER MAKING . . .

Texrope, Streambarker and *Hi-Density* are Allis-Chalmers trademarks.

ALLIS-CHALMERS

PERSONAL MENTION

Items About New and Old Tappimen

New TAPPI Members

Necati Buskurt, Superintendent of Pulp factory, Seluloz Sanayii Muessesesi, Izmit, Turkey, a 1940 graduate of Hochschule, Koethen, Germany.

Anton P. Bisschop, Managing Engineer, Verenigde Papierfabricken, Eerbleek, Holland, a 1949 graduate of Technische Hogeschoel, Delft, Holland.

Edward Blondeel, General Manager, Papeteries Generales Belges, Brussels, Belgium, a 1930 graduate of Ghent State University.

Roland R. Bultman, Chemist, Consolidated Water Power & Paper Co., Wisconsin Rapids, Wis., a 1940 graduate of St. Norbert College.

Kenneth E. Burns, President, Burns Mfg. Co., Aitkin, Minn., a 1936 graduate of Stanford University, with an MBA degree from Harvard in 1938.

Keith E. Bradway, Student, The Institute of Paper Chemistry, Appleton, Wis., a 1948 graduate of Purdue University.

Wilson S. Cathcart, Development Chemical Engineer, E. I. du Pont de Nemours & Co., Niagara Falls, N. Y., a 1948 graduate of University of Pennsylvania.

James O. Christensen, Sales Engineer, A. O. Smith Corp., New York City, a 1939 graduate of the University of Wisconsin.

Leslie M. Clark, Technical Service Manager, Imperial Chemical Industries Ltd., Alkali Div., Northwich, Cheshire, England, a 1929 graduate of Sydney Sussex College, Cambridge University, with a Ph.D. degree.

Robert T. Clark, Assistant to Chief Chemist, International Paper Co., Mobile, Ala., a 1947 graduate of the University of Alabama.

Nils Colliander, Technical Manager, A/B J. W. Enqvist O/Y, Lielahti, Finladn, a 1920 graduate of Helsingfors Institute of Technology.

Elmo L. Cooley, Groundwood Production Clerk, International Paper Co., Palmer, N. Y. Attended Queens College and the University of Pennsylvania.

Alfredo F. de Villa-Abrille, Manager and Supervising Chemical Engineer, San Miguel Brewery, Inc., Manila, Philippines, a 1938 graduate of the University of Sto. Tomas.

Wayne P. Draper, Technical Service Engineer, Hercules Powder Co., Wilmington, Del., a 1939 graduate of Colorado College of Agriculture & Mechanic Arts.

G. Milton Ellis, Jr., Superintendent, Power Dept., Southern Paperboard Corp., Port Wentworth, Ga., a 1937 graduate of the University of Alabama.

Esbjorn Eftring, Head of Cellulose Research Laboratory, Stora Kopparbergs Bergslags A/B, Falun, Sweden, a 1938 graduate of the University of Lund with a Ph.D. degree.

Johannes Ehlert, Technical Director, Comp. Melhoramentos de São Paulo, São Paulo, Brazil. Received Ph.D. degree from Heidelberg University, Germany.

Thomas N. Finical, Mill Manager, Sidney Roofing & Paper Co., Victoria, B. C., Canada, a 1933 graduate of Ohio State College.

Pinckney L. Frost, Manager of Sales, Innis, Speiden & Co., New York, N. Y.

Jack Grant, Vice-President, Chemipulp Process Ltd., Montreal, P. Q., Canada.

Leif Grogaard, Technical Director, A/S Union, Skien, Norway, a 1922 graduate of Trondheim College, Norway.

Jean A. L. Guay, Technical Salesman, Standard Chemical Co. Ltd., Montreal, P. Q., Canada, a 1945 graduate of the University of Montreal.

Thomas F. Hart, Associate Chemist, the Linde Air Products Co., Tonawanda, N. Y., a 1943 graduate of the State University of Iowa.

Charles W. Hayden, Sales Manager, Hayden Wire Works, Inc., Springfield, Mass. Attended Syracuse University.

Hans Heller, General Manager, Papierfabrik Weissenstein, A. G., Pforzheim, Germany, a 1933 graduate of Technical University Darmstadt, with a Ph.D. degree.

Philip L. Hovey, Mill Manager, Oxford Miami Paper Co., W. Carrollton, Ohio, a 1939 graduate of Yale University.

Hans Jenssen, Librarian, Food & Agriculture Organization of the United Nations, Washington, D. C.

Walter R. Keating, Control Superintendent, Quebec North Shore Paper Co., Ltd., Baie Comeau, P. Q. Canada, a 1936 graduate of the University of Toronto.

Mearl A. Kise, Director, Virginia Smelting Co., W. Norfolk, Va., a 1930 graduate of Lehigh University, with a Ph.D. degree from Yale University in 1933.

John G. Leech, Graduate Student, The Institute of Paper Chemistry, Appleton, Wis., a 1947 graduate of Pennsylvania State College.

Albert Lefeberre, Superintendent, Personal Products Co., Milltown, N. J.

Liboslav Masner, Director, Leather and Paper Research Institute, SVIT National Corp., Otrokovice, Czechoslovakia, a 1921 graduate of the Tanner School, University of Liege, Belgium.

Emil R. Padavic, Assistant Technical Superintendent, Container Corp. of America, Manayunk, Pa., a 1929 graduate of Marquette University.

William E. Phillips, President, William E. Phillips, Inc., Chicago, Ill., a 1919 graduate of Louisiana State University.

Robert T. Powers, District Engineer, National Aluminate Corp., Chicago, Ill., a 1943 graduate of Wabash College.

Rune Rydberg, General Superintendent, Storviks Sulfit A/B, Storvik, Sweden, a 1936 graduate of the Royal Institute of Technology, Stockholm.

Harry M. Spring, Jr., Chief Inspector, Mutual Boiler & Machinery Insurance Co., Boston, Mass. Attended Wentworth Institute.

Erwin L. Schmidt, Manager, Zellstofffabrik Waldhof, Wiesbaden, Germany, a 1920 graduate of Technical Hochschule, Darmstadt.

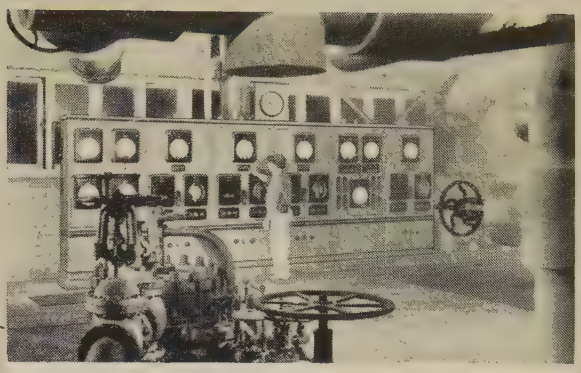
Ross D. Visger, Chemist, The Dow Chemical Co., Midland, Mich., a 1941 graduate of Central Michigan College.

Jack Watson, Manager of Technical Service, J. M. Huber Corp., Brooklyn, N. Y. Attended Cooper Union.

TAPPI Notes

Knute Amberg, formerly Technical Director of G. A. Serlachius O/Y is now Sales Engineer for O/Y Algol AB, Helsinki, Finland.

North.. Marathon Paper Mills of Canada, Ltd.
Marathon, Ontario



East.. Brown Company
Berlin, N. H.

West.. Weyerhaeuser Timber Company
Longview, Washington

South.. Mead Corporation
Kingsport, Tenn.

Better pulp production comes automatically with FOXBORO Digester Control

Wherever pulp is made, modern digester rooms like the ones shown above are proof that Foxboro Control is the shortest route to better operation.

With this advanced-design control, you can be sure that every cook will follow the exact schedule you want. Steaming and gas-off will be held to the requirements of the individual cook. Pulp quality and uniformity will be improved, with substantial savings in steam and chemicals.

Foxboro Control Instrumentation is available for *all* types of digesters. For detailed information, discuss your problems with the Foxboro engineer in your area, or write to **The Foxboro Company, 78 Neponset Ave.,** Foxboro, Mass., U.S.A.

FOXBORO
Reg. U. S. Pat. Off.

AUTOMATIC DIGESTER CONTROL

Arthur B. Anderson, formerly Chief Research Chemist for the Western Pine Association is now Research Biochemist, School of Forestry, University of California, Berkeley, Calif.

R. Ellsworth Annis, Jr., formerly Mechanical Engineer for Hollingsworth & Whitney Co., is now Plant Engineer, Cornell Wood Products Co., Cornell, Wis.

Robert S. Aries, President, R. S. Aries & Associates has moved his office from Brooklyn to 400 Madison Ave., New York 17, N. Y.

Robert E. Bauer, Package Engineering Consultant has moved his office from Chestnut St., to 1134 Land Title Bldg., Philadelphia 10, Pa.

Arthur C. Bird, formerly of Hardy S. Ferguson Co., is now Project and Electrical Engineer for Roderick O'Donoghue, 420 Lexington Ave., New York, N. Y.

Richard S. Buckley, formerly Chemist for Mohawk Paper Mills, Inc., is now with the Fernstrom Paper Mills Inc., Pomona, Calif.

Lloyd W. Carpenter, formerly Superintendent of E. W. Twitchell Co., is now Manager of the Paper Twisting Div., Plymouth Cordage Co., N. Plymouth, Mass.

John B. Chandler, formerly Sales Engineer for the Bristol Co., is now Manager, Pulp and Paper Division, Buffalo Pumps, Inc., Buffalo, N. Y.

Sidney M. Collier is now Asst. Superintendent, Puget Sound Pulp & Timber Co., Bellingham, Wash.

John D. Dickson, Jr., formerly Salesman, R. T. Vanderbilt Co., is now Sales Engineer in the Michigan-Wisconsin-Minnesota Territory for Stowe-Woodward, Inc., Newton Upper Falls, Mass. Mr. Dickson is located in Kalamazoo, Mich.

Russell F. Erickson is now Vice-President, Rayonier, Inc., Fernandina, Fla.

Donald M. Ferguson is now Director of Technical Service, St. Regis Paper Co., Deferiet, N. Y.

Hardy S. Ferguson is now Consultant for the H. K. Ferguson Co., 19 Rector St., New York, N. Y.

G. A. Franklin, formerly Control Superintendent, Booth Div., E. B. Eddy Co., is now in the Research Dept., Ontario Paper Co. Ltd., Thorold, Ont.

Uno G. Fryklund, formerly Mill Manager, Kramfors Co., is now Managing Director, Bowaters Svenska Tramassefabriker, Umea, Sweden.

Sakae Fukuyama is now Executive Director, Honshu Paper Mfg. Co. Ltd., Tokyo, Japan.

A. H. Gridley, formerly of Hardy S. Ferguson Co., is now Engineer for H. K. Ferguson Co., 19 Rector St., New York. N. Y.

Walker Hamilton, Jr. is now Sales Engineer for the Riegel Paper Corp., New York, N. Y.

George E. Jackson, formerly Asst. Superintendent, Gaspesia Sulphite Co. Ltd., is now Process Engineer, Rayonier, Inc., Fernandina, Fla.

J. O. Julson, formerly General Manager, BFD Div., Diamond Match Co., is now Mill Manager Pulp Div., Weyerhaeuser Timber Co., Springfield, Ore.

Charles R. Kise, formerly Manager, Economy Pumps, Inc., is now Sales Engineer, Equipment Engineers, Inc., Wynnewood, Pa.

Francis I. Kittredge is now Manager, Apparatus Sales Office, Industrial Customers Div., General Electric Co., Philadelphia, Pa.

Frederick E. Macy has established a sales agency at 1066 Drexel Bldg., Independence Sq., Philadelphia, Pa.

Russell A. McCoy, Jr. is now Vice-President, C. M. Guest Sons, Greensboro, N. C.

Samuel P. Moore, formerly of the Brunswick Pulp & Paper Co., is now Chemical Engineer, Scott Paper Co., Hoboken, N. J.

Felipe Mussi is now Chemical Director, Celulosa Argentina S. A., Capitan Bermudez, Argentina.

Juan E. Phillips, formerly of the N. Y. State College of Forestry, is now Dyestuffs Salesman, Imperial Chemical Industries Co. of Great Britain, Santiago, Chile.

B. K. Roecker is now General Superintendent, Pollock Paper Corp., Dallas, Texas, and Columbus, Ohio.

Robert Schulein is now Control Technician, Ashland Paper Mills, Inc., 220 E. 42nd St., New York, N. Y.

James T. Sheehy is now Vice-President, Group Harbor Div., Rayonier, Inc., Hoquiam, Wash.

W. L. Stafford, is now Development Supervisor, Virginia Cellulose Div., Hercules Powder Co., Hopewell, Va.

R. W. Stevens, formerly Mill Manager, Angelus Paper Box Co., is now a Consultant, 920 De La Fuente St., Monterey Park, Calif.

Wesley N. Stickel, formerly of the N. Y. State College of Forestry is Chemist, Texon, Inc., South Hadley Falls, Mass.

Robert F. Sylvester, formerly Chemist, Parsons Paper Co., is now Chemist, Robertson Paper Box Co., Inc., Montville, Conn.

David P. Thomas, formerly Instructor of Wood Technology, N. Y. State College of Forestry, is now at the University of Washington, Seattle, Wash.

Wayne Wheelhouse, formerly Technical Director, Sitroux, Inc., is now Chief Chemist, Cupples-Hesse Corp., 4175 N. Kingshighway Blvd., St. Louis 15, Mo.

John Willy, formerly Chemical Engineer, Protective Papers, Inc., is now Research Director Glas-Kraft Inc., Lonsdale, R. I.

Robert L. Yoder is now Pulp Mill Superintendent, Gulf States Paper Corp., Tuscaloosa, Ala.

* * *

M. D. Shriver, Buffalo, N. Y., has succeeded John B. Chandler as the official representative of the Bristol Co., Waterbury, Conn., in TAPPI.

The Western Precipitation Co., Los Angeles, Calif., has become a sustaining member of the Technical Association.

Industry Notes

John Waldron Corp., New Brunswick, N. J., has acquired the business of the Meadows Machine Works, So. Kearny, N. J. The present line of Waldron heavy-duty machines will be amplified by the much wider range of Waldron-Meadows medium-duty machines for coating, printing, embossing, laminating, and impregnating and supplementary equipment for converting operations on materials such as paper, textiles, plastics, foil, and film.

* * *

The General Electric Co., Schenectady, N. Y., advises that for the first time, a steam turbine has been applied to drive a log chipper. At the Fibreboard Products, Inc., mill at Antioch, Calif., a 2500-hp. turbine powers the chipper so that logs 20 ft. in length and 40-in. diam., are reduced to chips at the rate of one foot per second.

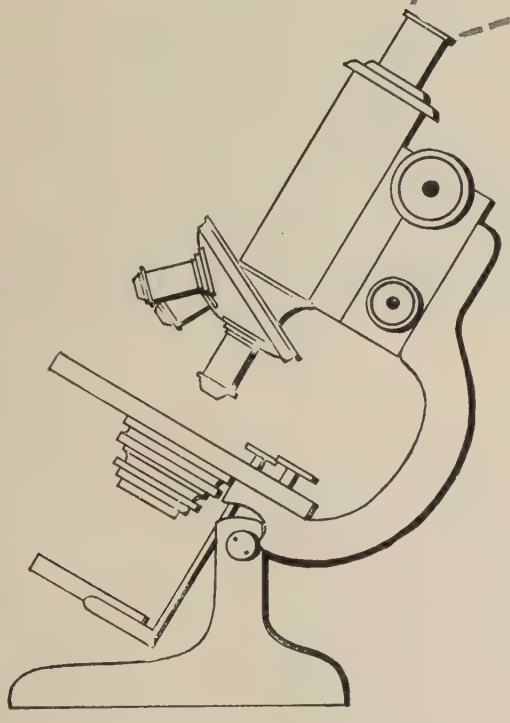
* * *

The Pusey & Jones Corp., Wilmington, Del., is now manufacturing Aerodyne dust and fly ash collectors for use in pulp and paper mills.

The basic unit of the dust collector system is a slotted sheet bent into the form of a cone. When a blower is started, the air or gas to be cleaned is passed at a high velocity over the filter surface and because of the design of slots in the cone, aerodynamic forces are set up. The resulting action which occurs deflects the particles away from the surface. This force keeps the dust particles suspended in the form of a thin cloud in

kill these pests...

Typical slime-forming organisms



with CHLORINATION by W&T

Quality conscious paper mill superintendents know from experience that chlorination, engineered to their stock or white water requirements by Wallace & Tiernan, effectively eliminates such slime-induced troubles as pin holes, slime spots, and poor sheet formation.

Such results—which have saved up to 400 dollars per day in many mills—are possible because chlorination kills the organisms that *cause* slime. It may be used alone on the fresh water supply, or together with chloramines or other corrective chemicals on the white water, for effective slime control in such key spots as the showers, screens, deckers, and at many other points.

Wallace & Tiernan Chlorinators are particularly effective in slime control work because they are especially designed from over 35 years' experience to give low cost, efficient performance.

For a survey on the chlorination needs of your mill without obligation, call your nearest W & T Representative now and start your plant on the road to slime free operation.

WALLACE & TIERNAN COMPANY, INC.

CHLORINE AND CHEMICAL CONTROL EQUIPMENT

NEWARK 1, NEW JERSEY • REPRESENTED IN PRINCIPAL CITIES

I-36

front of the slotted filter surface. The cleaned air escapes through the slots.

The suspended dust layer is carried at high speed to the outlet end of the cone where it is withdrawn into a secondary circuit along with a small portion of the air or gas. In the secondary circuit, the dust is precipitated and collected in a small container.

* * *

The Federal Security Agency has issued Supplement 213-Public Health Reports "A Statistical Summary of Sewage Works in the United States." This report summarizes, interprets, and analyzes data on the sewage works, based on the 1945 Inventory of Water and Sewage Facilities (Supt. of Documents, Govt. Printing Office, Washington 25, D. C., 10 cents).

* * *

A contract has just been signed by Messrs. Celdecor Africa (Pty.) Ltd. of Benoni, Union of South Africa, to design and erect a bagasse pulp and paper mill for Messrs. Refinadora Paulista S. A., São Paulo, Brazil. This mill will have the most modern features of the continuous process, and will take advantage of the experience obtained in the other two mills mentioned above, as well as in Celdecor-Pomilio pulp mills which are using cereal straw or espartograss, like the new mill of Messrs. Compagnie Nord-Africaine de Cellulose S. A. in Algeria, North Africa.

The bagasse is opened up and freed from pith by mechanical means. The cleaned material is blown to the top of the digester house, where it is impregnated with a weak caustic solution. The height of the digestion tower for bagasse can be the same as for straw, i.e., 50 feet.

The main feature of the Celdecor-Pomilio digestion towers is that, although open at the top, they cause a gradual increase in temperature toward the bottom portion owing to the hydrostatic pressure. The height of the tower depends on the type of material to be cooked and the degree of digestion required. The heat for the cooking is supplied only by means of steam jackets, so that all the steam used is returned to the boilers as condensate. There is no dilution of the liquor in the digestion tower, while the pulp travels down slowly and smoothly.

The bagasse will be evenly and very well cooked by the time it reaches the bottom of the tower, from which it is continuously extracted, and pumped to the wash filters.

The black liquor is washed out on Celdecor-Paxman vacuum filters. These filters were only introduced to the pulp industry about 5 years ago, when one was installed in the "Celdecor" pilot plant near London. They pick up the pulp at any consistency up to 5 per cent, and they deliver it nicely washed at up to 21 per cent, which is very good for the wetter pulps from straw, bagasse, etc. The filters are extremely easy to run, which is favorable as far as labor is concerned. The Celdecor-Paxman filters have a suction-drum which only dips into the pulp suspension for about 30 per cent of its surface, so that the remaining 70 per cent are available for displacement washing.

Following the usual lines of the Celdecor-Pomilio process for fine paper pulp, the digested bagasse will be chlorinated by means of chlorine gas coming straight from the electrolitic plant to be included in this scheme. Previously the pulp has to be dewatered in screw presses to about 30% consistency, opened up and fed to the gas-chlorination tower, which consists of a number of stoneware pipes. The chlorinated pulp is extracted from these pipes in a similar way as applied to the digestion tower, and the pulp is given a further chlorination in a retention tower. Next the pulp is washed and given a high-density alkaline treatment.

Finally the pulp will be screened, and bleaching will be done in a continuous Celdecor-Kamyr plant.

The mill will have ample supplies of good water. As it will be built next to a large sugar factory, the fuel efficiency of the whole scheme will be extremely favorable, and the cost of maintenance will be kept low.

* * *

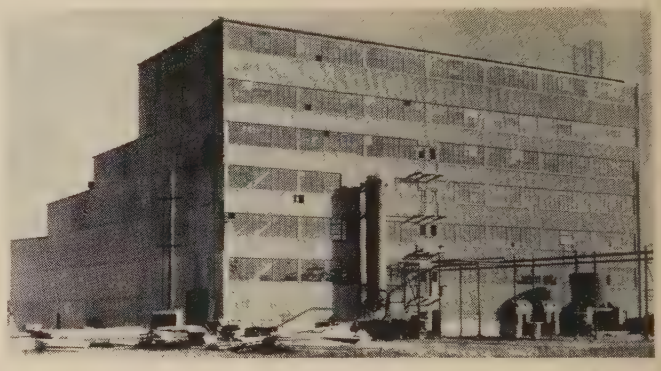
The American Cyanamid Co. advises that about 5 million dollars are spent annually in the work of its Stamford Research Laboratories. The laboratory floor space occupies 9.6 acres. About 800 people are employed, 400 of whom have technical degrees. It has 16,000 books in its library and more than 400 periodicals are received each month. More than 235,000 U. S. and foreign patents are kept on file. Six hundred of these have been issued to personnel of the laboratories. Among the products developed at Stamford are melamine resins (for wet strength papers), acrylonitrile (for synthetic fibers and rubber), parathion (a powerful insecticide), sulphadiazine, and Tagathen (an antihistamine for hay fever and asthma).

* * *

The Noble & Wood Co. was awarded the contract for 20 beater and machine chests for the Downingtown Paper Co., Downingtown, Pa. The chests are of Chemtile construction installed with individual motors and drives for each of the propeller agitators.

* * *

The Dow Chemical Co. announces that a new Midland, Mich., plant has begun large-scale production of powdered Methocel. This production represents the first quantities of



New Methocel plant at the Dow Chemical Co.

the material produced in powdered form on a commercial scale. Methocel or methylcellulose is a water soluble synthetic gum used as a thickener, binder, suspending agent, emulsion stabilizer or as a sizing or coating agent.

* * *

The Chicago Bridge & Iron Co., 332 South Michigan Ave., Chicago 4, Ill., has recently published an 8-page pamphlet on The Watersphere, an elevated water tank to provide gravity water pressure for general service and fire protection. The sphere is built entirely of steel plates with butt-welded construction. It is supported on a cylindrical column, the flared base of which rests on and is anchored to a circular concrete foundation.

* * *

The Arabol Manufacturing Co., adhesives manufacturers has opened a factory at 225 Moore St., S. E., Atlanta, Ga. Frank J. Michael is in charge of sales for the newly created Southeastern Division.

* * *

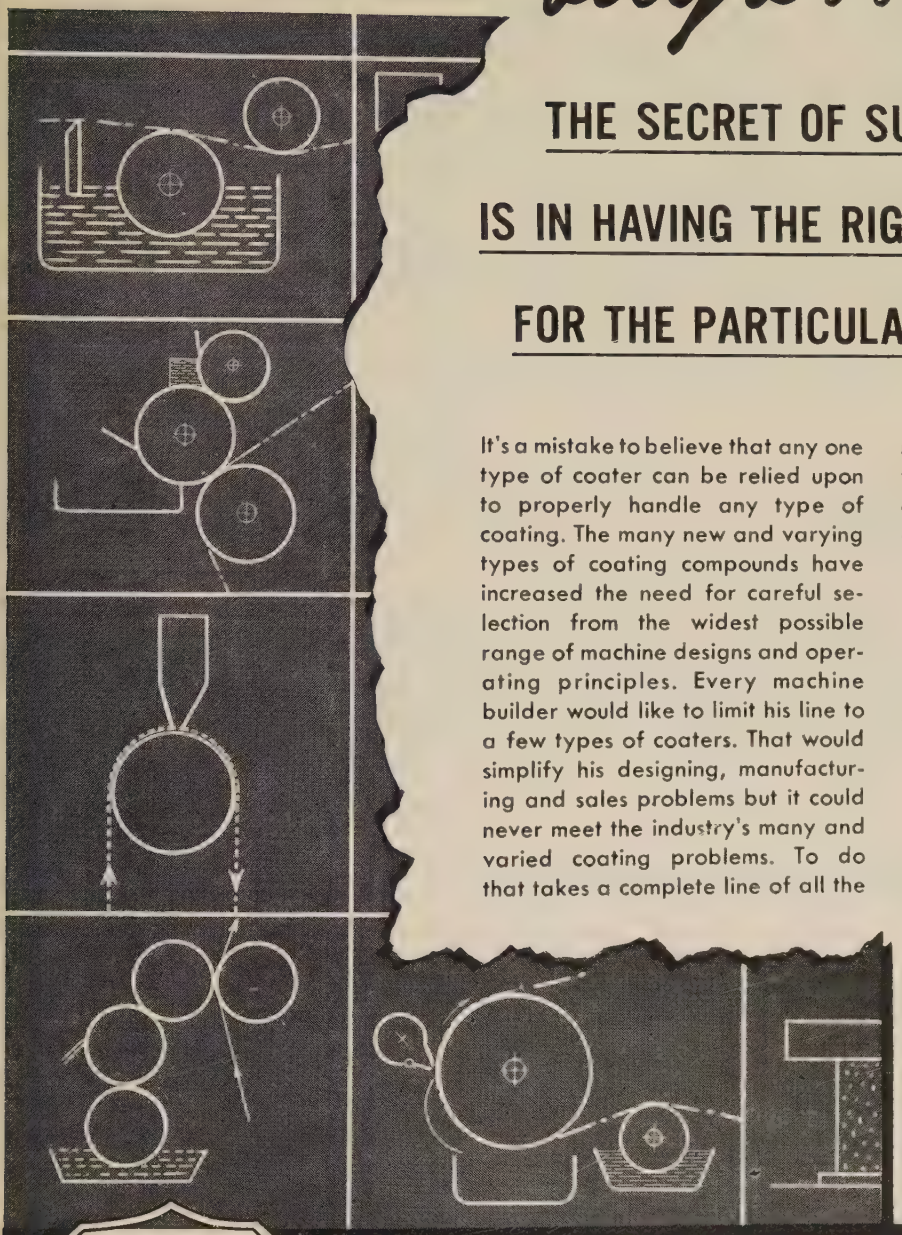
Hiroshi Eguchi, Chemical Engineer of the Kokusaku Pulp

Waldron says . . .

the industry's headquarters for converting machines

IF IT IS NEW—WALDRON HAS IT

THE SECRET OF SUCCESSFUL COATING IS IN HAVING THE RIGHT TYPE OF COATER FOR THE PARTICULAR TYPE OF COATING



It's a mistake to believe that any one type of coater can be relied upon to properly handle any type of coating. The many new and varying types of coating compounds have increased the need for careful selection from the widest possible range of machine designs and operating principles. Every machine builder would like to limit his line to a few types of coaters. That would simplify his designing, manufacturing and sales problems but it could never meet the industry's many and varied coating problems. To do that takes a complete line of all the newest types of machines — a line wide enough to cover every possible coating requirement — stock, type and weight of coating, speed, drying, etc.

The dependable WALDRON line of coaters offers just such a choice of machines, developed, designed and built by an organization with the longest experience in coating processes.

There's a WALDRON Coater best suited for your particular type of coating—and a laboratory test to prove it, if necessary.

The above applies to Laminating, Embossing, Printing and most other converting operations. To get the right machine for the specific purpose it will pay you to consult WALDRON first.

Coating Machines
Creping Machines
Embossing Machines
Floor Cov. Machines
Gumming Machines
Laminating Machines
Printing Machines
Rubber Mill Machines
Wall Paper Machines
Waxing Machines
Winders and Slitters
Special Design Machines

JOHN WALDRON CORP.

Main Office & Works

NEW BRUNSWICK, NEW JERSEY

BUILDERS OF QUALITY MACHINES SINCE 1827

Industry Co. Ltd., makers of rayon pulp in Japan has been awarded the 1949 Harold R. Murdock Prize. Mr. Murdock who is with Robert and Company Associates, Atlanta, Ga., and is the donor of this prize, advises that the award this year consisted of four books "Chemical Engineers Handbook," by Perry, "Wood Chemistry," by Wise, "Chemistry of Pulp and Paper Making," by Sutermeister, and the "Chemistry of Cellulose," by Heuser. The books were presented to Mr. Eguchi on April 27 at a meeting of the Japanese TAPPI held in Tokyo. The award is made annually by Mr. Murdock to encourage a scientific approach within the Japanese pulp and paper industry. The candidate must have presented a dissertation on any subject dealing with the industry at a regular meeting of Japanese TAPPI and published in its proceedings.

* * *

The Dorr Company, Inc., Barry Place, Stamford, Conn., has issued two interesting bulletins. An 8-page bulletin entitled "Dorrco Flash Mixers and Flocculators," describes equipment used primarily for the pretreatment of industrial water supplies. A 12-page bulletin entitled "Dorrco Continuous Recausticizing System for the Alkaline Pulping Process," describes the recovery of soda chemicals, the production of clarified green liquor, washing the green liquor dregs, and removing and washing the lime prior to burning.

* * *

Howard J. Soriano has been appointed Eastern Sales Manager for Sinclair & Valentine Co. Mr. Soriano has been with the company for fifteen years.

* * *

Link-Belt Co., 307 N. Michigan Ave., Chicago, Ill., has just issued an 8-page pamphlet "Automatic Controls Book No. 2349" announcing a new line of automatic speed controls for the Link-Belt P.I.V. variable speed drive.

* * *

Oakite Products, Inc., 115A Thames St., New York 6, N. Y., has issued a 14-page booklet on "How to Strip Paint." It gives an interesting account of materials and procedures designed to simplify the removal of oil-base and synthetic paints, enamels and similar finishes from metal surfaces.

* * *

Clark & Vicario Co. have removed its offices from New York City to 81 Pondfield Road, Bronxville, N. Y.

OBITUARY

Louis S. Keyser

Louis S. Keyser, Chemist at the Central Chemical Laboratory of Rayonier, Inc., Shelton, Wash., died on June 7, 1950.

Dr. Keyser was born at Ancon, Panama Canal Zone, on December 20, 1910, but spent most of his life in the Northwest, receiving his B.S. degree in Chemistry from the University of Idaho and his M.S. degree a year later. He received his Ph.D. degree at the University of Illinois in 1938. From 1938 to 1940 he was a Research Fellow at the University of Minnesota. He joined the Rayonier, Inc. Technical Staff in 1940.

SLIMICIDE
COMPOUND G-4*
(2, 2'-dihydroxy-5, 5'-dichloro diphenyl methane)
EFFECTIVE, NON-TOXIC, ECONOMICAL
*Trade-Mark
SINDAR Corporation
Industrial Aromatics and Chemicals
330 West 42nd Street, New York 18, N.Y.
Branches: Philadelphia • Boston • Cincinnati • Detroit • Chicago • Seattle • Los Angeles • Montreal • Toronto

IF YOU NEED A POWERFUL OXIDIZING AGENT

...Mathieson

$NaClO_2$

130% AVAILABLE CHLORINE

ADVANTAGES:

- Bleaches cellulosic fibers without attacking and degrading the fibers.
- Extremely soluble in water.
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LOCAL SECTION ACTIVITIES

Reports of Meetings, Personnel and Events

Pacific Coast Tri-Association Meeting

An interesting innovation in the annals of the pulp and paper industry on the Pacific Coast occurred on May 11, 12, 13, 1950 with the first tri-association meeting at the New Washington Hotel in Seattle. As the name implies, the meeting was sponsored jointly by the Pacific Section of TAPPI, The Pacific Coast Division of the American Pulp and Paper Mill Superintendents Association Inc., and the Pacific Coast Branch, Technical Section of the Canadian Pulp and Paper Association.

The idea of a three-way joint meeting of the three groups developed from a similar joint affair between the Canadian Technical Section and the Superintendents Association held in Victoria, B. C. in May, 1949. A record attendance in Seattle of over 450 indicates that the meeting "took" with the industry and points toward the continuance of the "WIMPPI" plan in the future (WIMPPI = Western International Meeting Pulp and Paper Industry).

A. C. McCorry, St. Regis Paper Co., Tacoma, was General Chairman of the meeting and was ably assisted in planning and staging the meeting by R. I. Thieme, Soundview Pulp Co., Everett, L. R. Hartman, Weyerhaeuser Timber Co., Pulp Division, Everett, D. J. McLaurin, Powell River Co., Powell River, B. C., who arranged the Technical Program; F. R. Armbruster, The Dow Chemical Co., Seattle, Hotel and Financial arrangements; Gordon B. Anderson, Puget Sound Sheet Metal Works, Seattle, Registration and Housing; W. M. Clines, General Chemical Co., Seattle, and W. A. Salmonson, Draper Bros. Co., Portland, Entertainment; Joe McQuaid, Electric Steel Foundry Co., Seattle, Golf; R. M. True, General Dyestuff Corp., Portland Mailing; Carl Castle, The Dow Chemical Co., Seattle, Printing and Publicity.

A résumé of the Technical Program follows:

L. R. Hartman, Weyerhaeuser Timber Co., Pulp Division, Everett, Wash. acted as Moderator of the session held on Friday morning May 12.

(1) "The Camas Paper School" by Walter C. Jacoby, Crown Zellerbach Corp., Camas, Wash. In this paper Mr. Jacoby discussed the unique features of the Camas school which offers a comprehensive four year course in pulp and paper technology. Credits earned in this school are honored by leading colleges and universities of the Pacific Northwest.

(2) "Preconditioned Paper" by H. R. Russell, Everett Pulp & Paper Co., Everett, Wash., was an interesting account of the operation of a preconditioning chamber through which paper from the paper machine is passed before going to the supercalender. Accurate control of temperature and humidity in the two sections of the conditioning chamber results in a stabilized moisture content in the finished paper and the sheet shows a minimum tendency toward dimensional change in subsequent finishing and printing operations.

(3) In his paper "Duties and Responsibilities in the Pulp and Paper Mill" E. P. Barthelemy, Crown Zellerbach Corp., Camas, Wash. discussed the many factors involved in the smooth-running mill. His ideas on job training, foreman responsibility, and frequent conferences between the supervisor and his crew were well received by the audience.

Friday afternoon was given over to mill visits and golf. A large group visited the mills in Tacoma and Everett for an interesting view of new equipment and processes.

The technical sessions resumed on Saturday, May 13, with R. I. Thieme, Soundview Pulp Co. presiding at the morning meeting. Two papers were presented at this session both of which will be published in *Tappi* and are given here by title only.

(1) "An Analysis of Some Sulphite Waste Liquor Processing Schemes" by Dr. J. L. McCarthy, Dept. of Chemistry and Chemical Engineering, University of Washington, Seattle, Wash. This paper is one of the series produced under the Pulp Mills Research Project at the University of Washington.

(2) "The Magnesium Oxide Sulphite Waste Liquor Recovery Process" by Svarre Hazelquist, Weyerhaeuser Timber Co., Pulp Division, Longview, Wash. and Charles Rogers, Babcock & Wilcox Co., New York, N. Y.

At the men's luncheon following the technical meeting, Toastmaster John Guthrie, British Columbia Pulp & Paper Co. Ltd., Woodfibre, B. C., introduced Dr. Raymond B. Allen, President of the University of Washington who addressed the group on current economic and political problems.

The concluding technical Session was held Saturday afternoon with D. J. McLaurin, Powell River Co. Ltd., Powell River, B. C., presiding as Moderator. Three papers were presented which will be published subsequently in the trade magazines.

(1) "The Performance of Cowan Screens on Western Groundwoods" by A. R. Ferguson, Pacific Mills Ltd., Ocean Falls, B. C.

(2) "Heat Transfer from Hot Moist Air in a Spray Tower" by D. L. Stewart, Powell River Co. Ltd., Powell River, B. C.

(3) "Current Research in the Division of Cellulose Chemistry at McGill University" by C. B. Purvis, Professor, Industrial and Cellulose Chemistry, McGill University, Montreal, P. Q.

The TAPPI Pacific Section held its annual business meeting on Saturday May 13. Officers elected for the year 1950–1951 are

Chairman—R. I. Thieme, Soundview Pulp Co., Everett, Wash.
Vice-Chairman—E. O. Ericsson, Puget Sound Pulp & Timber Co., Bellingham, Wash.
Secretary-Treasurer—R. M. True, General Dyestuff Corp., Portland, Ore.
Members of Executive Committee: F. J. Weleber, Publishers' Paper Co., Oregon City, Ore.; S. E. Hazelquist, Weyerhaeuser Timber Co., Longview, Wash.; W. A. Salmonson, Draper Bros. Co., Portland, Ore.; H. W. Bialkowsky, Weyerhaeuser Timber Co., Longview, Wash.; W. F. Holzer, Crown Zellerbach Corp., Camas, Wash.; and J. M. McEwen, Weyerhaeuser Timber Co., Everett, Wash.

Ladies attending the Joint Meeting were entertained at a luncheon and theatre party. A banquet and dance Saturday evening concluded the first successful WIMPPI which seems destined to become a tradition on the West Coast.

R. M. True, *Secretary*
Pacific TAPPI

Lake States

The Lake States Section met at the American Legion Club House, Appleton, Wis., on Tuesday, May 9.

John P. Gerhauser of the Appleton Wire Works, Appleton, Wis., gave a talk on "Fourdrinier Wires."

B. F. Shema of the Institute of Paper Chemistry discussed "The Microbiological Degradation of Woolen Felts."

Chicago

The Chicago Section met at the Chicago Bar Association Quarters on May 17. The following officers were elected to serve during the ensuing year:

Chairman—Albert K. Roach, Wm. H. Welsh Co., Inc., 2241 Indiana Ave., Chicago 16, Ill.
Vice-Chairman—Robert Ragan, Chicago Carton Co., 4200 So. Pulaski St., Chicago, Ill.
Secretary—Arthur A. Duetsch, Container Corp. of America, 900 Ogden Ave., Chicago, Ill.
Treasurer—Charles H. Krebs, Atlas Boxmakers, Inc., 5025 W. 65th St., Chicago, Ill.

R. G. Macdonald, Secretary-Treasurer of TAPPI was present and outlined the current activities of the national Association.

Talks on various aspects of "Adhesives" were given by Robert L. Pett, Resyn Technical Service, National Adhesives Div., National Starch Products, Inc., Samuel Shuller, Technical Director, Paisley Products, Inc., and Roger Shoals, Manager of the Adhesives Dept., Stein-Hall & Co., Inc., all of Chicago.

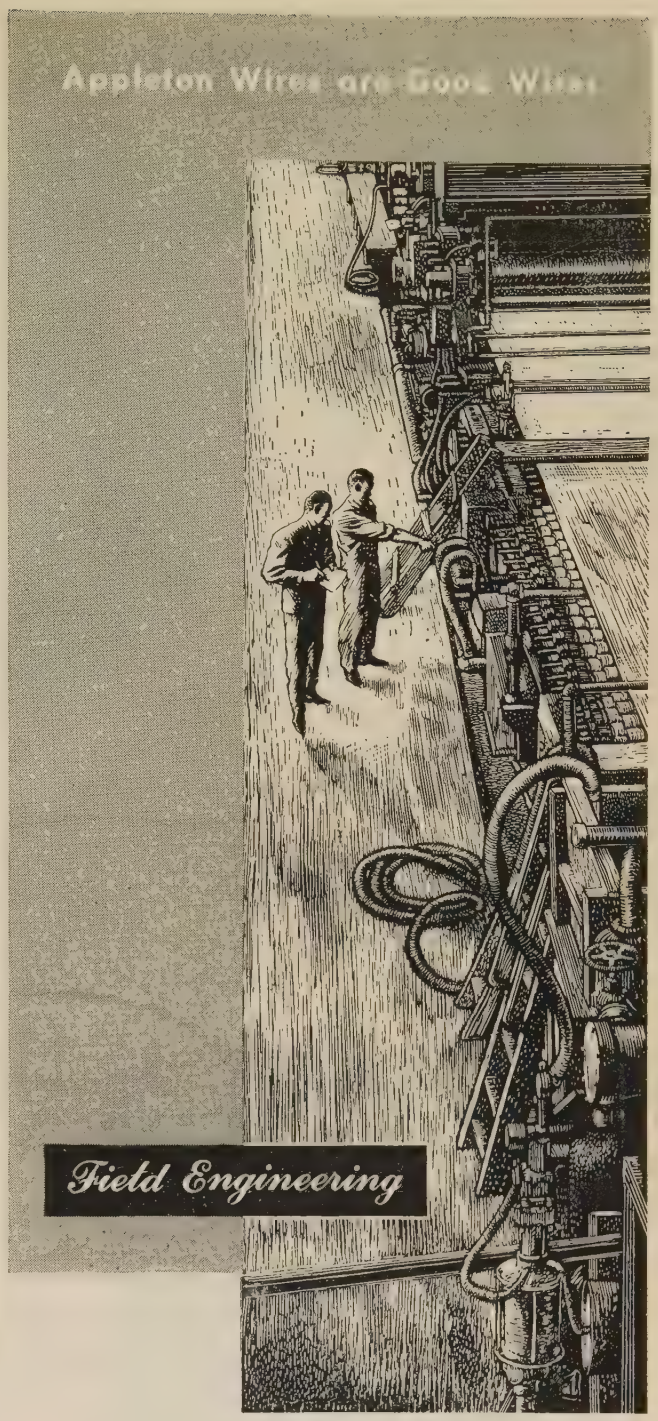
The remarks of these speakers follow:

Remarks of Mr. Pett on Resins

"The adhesive industry has undergone a great many changes in the past 15 or 20 years and I am sure that you will agree with me that the innovation of synthetic resin adhesives is the most significant. If this subject of resin adhesives was being discussed 15 years ago, the subject could be covered most thoroughly in 5 or 10 minutes. However, this field has grown and expanded so that it would be impossible to cover it in the short time allotted me at this meeting. For this reason I will attempt to give you a general outline of the most significant uses and developments in the synthetic resin field to date.

"The use of starch and dextrine adhesives proved satisfactory for many, many years in the packaging field, however, as the packaging field grew and began to play a more and more important part in industry, we found that these adhesives were not adequate. With the development of cellophane sheeting, plastic films, improved coatings for paper, and high-speed packaging machines the adhesive industry was forced to look for other types of base raw materials to be used in the formulation of their adhesives. After exploring many fields we finally found that the synthetic resins showed the most promise.

"The first synthetic resin adhesives were developed for use by cellophane converters. These were the solvent lacquer adhesives. The cellophane industry quickly adopted them for use in the manufacture of cellophane bags and laminations of cellophane to various surfaces. These lacquers proved quite satisfactory in most respects. However, they were fairly slow drying and the concentration of solvents often caused bleeding of inks. We took this problem under consideration and came up with the resin emulsion type adhesive. We have found that emulsions show excellent flexibility of formulation. Generally speaking, emulsion adhesives consist of a resinous base material dispersed in water, a plasticizer, and often times a solvent. Simply by changing the ratio of resin and plasticizer these adhesives can be made faster or slower; harder or more flexible. Solvents are added to give penetration into special lacquer coatings on paper, or to penetrate films such as cellulose acetate or cellophane. Emulsions are fast setting because as soon as they come in contact with a surface the emulsion breaks. That is, the water penetrates the surface and the solid resin remains to bond the surface together. It is for this reason that we advocate the use of a very thin film of adhesive. The most important factor in the rapid use of emulsions is that once the film has dried it cannot be re-emulsified by water. It is this characteristic that gives the adhesive its high degree of water resistance. There are several large folding box manufacturers today who are adopting the use of these emulsions for just this reason. Even though the adhesives are more expensive than starch and dextrine types they feel the added protection from moisture and the increased speed of application will make up for the added cost. Along this same line of reasoning the manufacturer has to be sure that even though the adhesive seems to be holding in his plant it must remain that way for long periods. Starches, dextrines, and animal glues tend to dry out and crystallize while the resin emulsions remain flexible and do not soften under high humidities and temperatures.



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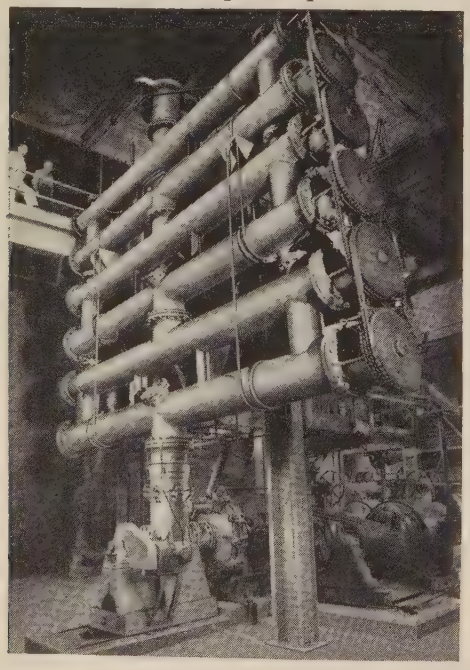

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"With the advent of World War II the packaging field was called upon to perfect better and more protective carriers for war supplies. This called for stronger packages and packages that would withstand submersion in water, the elevated temperatures of the tropics, and the subzero temperatures of the Arctic. Naturally the adhesive industry had to develop the adhesives to seal these packages. Adhesives were developed that would withstand water immersion for several days and also protecting coatings for markings on these packages. At the end of the war the packaging industry was quickly converting to peace time operations and were clamoring for more and more synthetic resin adhesives. They found that to meet competition it was necessary to turn out packages that were more versatile and gave the best protection.

"In the fiber can and tube industry, for example, there was a need for cans and tubes that were more water resistant, tougher, and better moisture-vapor barriers. At the present time we have synthetic resins that will increase production. "Resyn" adhesives today will wind tubes at a faster rate than was thought possible several years ago. These synthetic resins are also being tested today to be used to eliminate barrier materials such as parchment and foil which are now used at great expense to get the desired moisture-vapor resistance. Along with the lacquer and emulsion adhesives, we have developed the hot-melt type of adhesive. A hot melt is in a solid state at room temperature and must be heated in order to get it to a fluid consistency. These hot melts contain neither solvents nor water and have 100% solids. Hot melts are well adapted for use as protective coating and for applications where immediate setting time is necessary. A film of hot melt applied to a surface sets as soon as it hits the air. These hot melts can be made to form hard lustrous finishes or more flexible films.

"Perhaps the newest and most important class of adhesives developed to date are the heat seals. We have developed heat-seal adhesives in the three classes of resins mentioned; i.e., emulsions, lacquers, and hot melts. The heat-seal adhesive can also be subdivided into the direct contact and delayed tack type. The direct contact heat seal is coated on a surface and allowed to dry. At a later time this surface is brought into contact with another surface and heat is applied. A bond is affected instantly. The delayed tack type is coated and allowed to dry, but it is reactivated with heat and develops a high degree of tack immediately. It holds this tack for some time. The advantage of this delayed tack lies in the fact that the two surfaces do not have to be in contact when heat is applied. A delayed tack adhesive finds use in the bottling industry where the label is heated and then placed on a bottle. The direct contact heat-seal type finds uses in fields such as gummed tape, jewelry boxes, and in manufacturing milk cartons.

"At the present time most of these heat-seal adhesives are of the emulsion type. However, we have developed lacquer types which are particularly adopted for bonding coated surfaces, foil, and cellophane. Along these same lines there are hot melts which are used as heat seals. The hot melt is well adopted for use by manufacturers who coat rolls of paper and then print and cut them into labels. Another large field for hot melts is in the potato chip bag industry. Here the adhesive is applied to the bag in the bag plant and the potato chip manufacturer fills the bag and merely heat seals it to effect a closure. We feel that heat-seal adhesives will play a large part in the packaging field of the future. These adhesives can be applied to various surfaces and stored away for a period of time before using. When the converter uses these coated surfaces he can turn out a finished product at a very fast rate. He also eliminates the many problems of gluing that arise in the plant.

"The chemical industry has been making rapid strides in developing new transparent plastic sheetings. They began with coated glassines and cellophane. Now they have progressed to sarans, pliofilm, and polyethylene. Glassine and cellophane can be adhered without too much trouble, but we have had quite a time with the latter films. We now have adhesives for saran and pliofilm which give excellent adhesion. Our real problem has been the sealing of polyethylene. Polyethylene is resistant to alkali, acids, and up until now most adhesives. We have finally overcome this difficulty however, and we have adhesives that will bond polyethylene to kraft, foil, and to itself. Polyethylene is not being used in very great quantities at the present time but we feel that it has great potentialities. This fact is borne out by the research programs being carried out by the large converters and by all of the adhesive manufacturers. While polyethylene adhesives are not perfect yet, they are being improved every day.

"Other fields requiring resins are water-resistant foil laminations and coatings for inked surfaces. Many breweries are using foil-laminated labels to give their bottles a more appealing appearance. Naturally a water-resistant adhesive is needed and several resins have been developed for this application. For many years converters have been laminating acetate to printed board but recently there has been need for laminations of acetate to foil, pliofilm, and vinyl sheets. Several of these laminations are being made today and the outlook for the future is for more

and more of these combinations. Along these same lines there are many manufacturers who are attempting to coat printed and plain surfaces with materials to replace acetate laminations. Several of these experimental coatings are being tested in various plants and we should have the answers in a short time. These coatings will not only give a high glossy finish but they will also be greaseproof and water resistant."

Remark by Mr. Schuller on Animal Glues

"Animal glue is undoubtedly, the oldest adhesive material known to mankind. Today it still earns the high regard of adhesive technicians despite the publicity given to the glamorous new adhesive materials such as synthetic resins and plastics, lattices, and the like. There is no current indication that animal glues will be supplanted in those packaging operations where it is now established as no synthetic adhesive material has ever been developed which contains all the useful characteristics of animal glues at a comparable cost. The use of animal glues in packaging adhesive operations, parallels the development of that industry from the very beginning. Animal glue adhesives when made into compounded, nonwarp, flexible cake form have benefited from the adhesive industry's research in the last ten years and has become a part of the scientific exploration into better, more economical ways of holding things together.

"Before discussing the finished forms of animal glue used by the package manufacturer, it will be necessary to review the basic raw materials from which they are made. Animal glues are prepared from bones, tendons, ligaments, and skins of animals. The chief ingredient of animal glues is collagen; a gelatin-like protein substance, amorphous, and somewhat flexible when dry. Being composed of long chain molecules, it exhibits many of the rubber-like and elastomeric qualities sought in newer synthetic resins and plastics.

"Animal glues in commercial use are divided roughly into two classes: hide glues and bone glues. Hide glues are prepared chiefly from tanners' hide trimmings. Bone glues can either be extracted from green bones or dry bones. Hide glues are considered the better adhesives, dry bones the next best, and green bone glue, the lowest quality of all. Since animals are not raised for the animal glue content, the supply and demand of these hides and leather control the supply and the price of the glue, since it is only a byproduct of the meat packing and tanning industries.

"All animal glues swell in cold water, dissolve in hot water and form a jelly on cooling. Chemically, they are a member of the colloidal group of adhesive materials. Steady advances in colloidal chemistry through research, has continually improved the compounding technique of adhesive manufacturers and the cake form glues available today, greatly out perform the adhesives of yesterday. Their ability to liquify under heat and solidify or jell on cooling, makes animal glues valuable for many industrial and packaging operations. By adjusting the speed of setting of the glue to the speed of the production operation, a smooth, harmonious performance can be achieved. Cake form animal glue in nonwarp and flexible grades are the accepted method of regulating the performance, characteristics to industrial requirements. This has been carried to a high degree of specialization as formulas are produced which take into consideration the changes in weather, seasons of the year, in order that optimum setting speeds can be realized. The timing of flexible glue film set and tack to conveyor-line speeds, machine rotation are production phases within prearranged margin of safety limits, is but one quality which keeps animal glues in tune with modern industrial and package manufacture requirements.

"Animal glues are evaluated from their qualities of jelly strength, viscosity, melting point, and setting point. Other qualities important to packaging adhesive use, include clarity, odor, and chemical contents. Both nonwarp and flexible glues are compounded from hide and bone glues by blending the various plasticizing materials, coloring ingredients, essential oils, defoaming agents, and preservatives. The selection and blending of animal glue grades, due to the wide variety of their test and origin with proper proportions of other ingredients to obtain the particular physical property in the end product, requires a chemical skill of high order. The modern adhesive chemist is a believer in specialization of compounding a formula that will fit the particular package manufacturer, labeling, or sealing operation with highest efficiency rather than by compromising to achieve interchangeability.

"Nonwarp glues are usually supplied to the trade in small cakes; six to ten pounds each, scored for separation. The purchaser usually melts up the cakes in premixers with measured amounts of water. Then the hot diluted glue is transferred to the glue reservoir on the machine. The largest packaging use of nonwarp glues is in the set-up paper box industry, where the lingering tack of the setting glue film enables quick adhesion of the label turn-in as the box shell is tightwrapped with the glued off outer label. Thin film formation, absence of foam, smooth

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coverage, quick but lingering tack, are the requirements for this work to gain highest production efficiency.

"Nonwarp glues and flexible glues are also obtainable with special chemical properties such as for use in lining and covering silverware chests, instrument cases and the like. For this work, the animal glues are specified with sulphur content less than 0.0008% and a sodium chloride content less than 0.05%.

"At this point, I would like to list some of the uses of nonwarp and flexible glues in various packaging operations:

USES OF NONWARP AND FLEXIBLE GLUES IN PACKAGING OPERATIONS

Containers, Paper
- Folding boxes (side seam, final closure).
- Set-up boxes (covering, tightwrap, hinging).
- Spiral tubes and cans (winding, labeling, capping).
- Convolute tubes and cans (winding, labeling, capping).
- Heavy cartons (laminating, hinging, interior construction).
- Envelopes and special bags (seams and bottoms).

Containers, Special, Christmas, Dual Purpose
- Leather covered boxes (laminating, covering, embossing, hinging).
- Canvas covered boxes (laminating, covering, embossing, hinging).
- Toy trunks.
- Jewelry boxes, metal, plastic combinations.
- Cosmetic containers, paper, wood, etc.

Containers, Wood
- Cabinets, silverware (wood joining, lining).
- Boxes, pencil (lining, covering).
- Sewing kits.
- Cigar boxes, humidor chests (wood joining, covering).

Advertising Displays, Counter Cards, Giant Packages
- (Printing, mounting, easel backing, bindery operations).

Miscellaneous
- Sizing for moulded pulp containers.
- Artificial flower, foliage manufacture.
- Flocking, glitter, tinsel application.
- Labeling of boxes, cans, bottles, etc.
- Plastercast molds

"I will now list some of the advantages and disadvantages of non-warp and flexible glues:

ADVANTAGES OF NONWARP AND FLEXIBLE GLUES

1. High margin of safety in bending strength.
2. Enables formation of thin films.
3. Minimum of absorption into board or paper.
4. Sets fast by cooling.
5. Enables high concentration of solids.
6. Exceptional smooth-machining properties.
7. Forms a continuous, cohesive film.
8. Freedom from crystallization.
9. Compatible with a wide range of plasticizers, chemicals to achieve specific operating requirements.
10. Stands much handling abuse
11. Low water content.
12. Rapid formation of strong initial bond.
13. Handles high-speed operations, hand or machine.
14. Holds to a wide range of packaging materials, paper, paperboard, leather, fabrics, wood, glass, transparent sheetings, porous and nonporous surfaces.

DISADVANTAGES OF NONWARP AND FLEXIBLE GLUES

1. Must be used hot.
2. Not all animal glues are odorless.
3. Cannot hold to paraffin, waxed surfaces, varnished, or pyroxylin-coated materials.
4. Unsuitable for excess moisture or refrigeration conditions.
5. Fast set not advantageous in intermittent operations.
6. Higher cost than many starch and dextrine adhesives.
7. Bond strength often in excess of requirements.
8. Time loss awaiting melting or heating up.

"I have attempted to show how animal glue is a very versatile base, from which we compound many of our finished adhesives. It has many advantages and some draw-backs. Whether we use animal glue as a base for some particular adhesive, depends a good deal on the adhesive chemist carefully weighing its properties against the properties of other types of adhesives such as you heard here tonight."

Remarks by Mr. Shoals on Dextrines

"This is my first experience with TAPPI. I am pleased with the opportunity. I have heard a great deal about your organization from some of my associates at Stein, Hall & Co. Their activities have brought them into closer contact with you than have mine. When I started my career in the adhesive business

one of the first words I heard was tapioca. Frequently the word was used in an abbreviated form—Tap. When I first heard of TAPPI, I groped briefly for some connection between tapioca and TAPPI; but I soon learned.

"My subject tonight is to be Adhesives—adhesives that are made from various types of carbohydrates. As you all know, this is an extremely broad subject and it covers many different fields. Indirectly, it is very closely associated with the paper industry because after you make the paper we have to glue it together in the various forms that people desire it, whether it be paper bags, boxes, envelopes, cartons, or whatever it may be. In doing this job prior to the war we had only three basic materials to work with, dextrines made from starch, animal glue, and casein. This was not too much to work with but we improvised pretty well and not too many jobs had to be shelved for the lack of an adhesive. Today, of course, we have many new commodities in the field of resins to work with that are extremely helpful to us in obtaining results that we never could have obtained before.

"I will leave all reference to this class of adhesives to my good friend, Dr. Pett. I shall confine my talk to dextrines. Dextrines are still the basis for most of the adhesives that are used in this country today. No doubt you all know the story of how dextrine was discovered. The story follows very closely the tale of Mrs. O'Leary's cow and the Chicago fire. Some say it was a textile mill in Dublin, others, a starch warehouse in London, but all are agreed that as a result of a fire it was discovered that starch in the presence of heat reduced itself to a gummy mass. After this discovery, further work was done and British Gum was made, and is still being made, being used extensively for many purposes. Additional work was done with starch and it was found that the acidulation of starch accelerated the conversion and made products of lower water taking properties, of light color, fast tack, and remoistening qualities with fair stick.

"Unfortunately, nothing that we make from starch sticks as hard as starch does; everything that we do to starch takes away stick. I think it will surprise you gentlemen who are so technically minded and who have contributed a great deal to the advances in the technique of making paper to learn that the dextrine industry has not progressed as much as we would like to think it has. The dextrines that are made today do not differ too much from the dextrines that were made fifty years ago. True, we know how to apply certain principles to dextrine much better now than we used to. Take for instance the Envelope Industry, they still have pretty much the same problems in the Envelope Industry today that they had thirty-five years ago. Dextrines normally will handle about 85% of all the standard paper for front seal work but that remaining 15%—thirty-five years ago had trouble with it—the adhesive manufacturers still have trouble with it. This paper, of course, is the rag content bond paper. This remark might bring some comment from my fellow competitors, but I think if anyone asked them a direct question- "Do you have a bond gum that will stick all bond papers for front seal work?" their answer would be just the same as mine, 'No.'

"The technique of converting dextrine has vastly improved, in fact it has even been converted with electronics, whereby the conversion takes place instantly. But again, I repeat this does not materially affect the end product that is produced. Today we manufacture dextrines, designating them by a name or a number, and some of these products throughout the years have become very well known and are automatically associated with a certain manufacturer. The specifications for all dextrines are relatively simple and include color, viscosity, pH, sugar content and solubility, but in order to obtain all of these qualities it is necessary to jockey formulas around quite often to continue to produce uniform products. And, in my mind, gentlemen, the most important thing that we have to do is to make uniform products. This is not as easy as it sounds. The dextrine manufacturers leave no stone unturned to accomplish this.

"It might interest you to know that the most common materials used to make these products are corn, starch, and tapioca flour. There is some potato and sago used, but corn is by far the largest produced. There are approximately 74 million pounds of corn dextrine manufactured annually for the adhesive and paper industries and in order to make this, there are 225 million bushels of corn used. In the over-all corn starch business there are 668 million pounds of starch manufactured annually. For every 100 pounds of starch that are manufactured 3 bushels of corn are required.

"Tapioca is imported into this country in substantial quantities but all of the tapioca imported is not used by the adhesive manufacturers. A great deal is used by the Food Industry and only the very top grades are used in the manufacture of tapioca dextrine, and these dextrines must have built into them certain characteristics for the most exacting type of work. At the present time most of our tapioca is coming from South America and in the last few years they have improved the quality of their flour greatly. It might also interest you gentlemen to know that prior

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to the war all the tapioca that we imported came from the Dutch East Indies, and at the time of the Pearl Harbor disaster our supplies were cut off just like you would turn off a running faucet. One day we had an ample supply the next day we had none. This had about the same effect on us as if tomorrow they said to you paper manufacturers, "Gentlemen, there is no more pulp." This left our industry in a chaotic condition because we had been firm in our belief that in order to make the top quality dextrine for the type of work as we specialize in, tapioca was a must. I think the adhesive manufacturers, as a whole, did a splendid job in keeping all industry running at wartime capacity with improvised material. Not only did we do a good job for industry but it caused many die-hards to change their viewpoint on many things that apply to the manufacture and use of dextrine and adhesive products.

"We still cannot report to you that we are out of the woods as far as tapioca is concerned. We have received some token shipments from Java and we expect more as time goes on. Within the next year or two we should be back in business on tapioca. This will help us a great deal, as industry is calling on us now to produce better products to meet their demands for increased production with less help. This demand is brought about by the fact that they must produce more with less people in order to remain competitive due to increased labor cost.

"The Packaging Industry started this by asking the machine builders to construct faster machines. This they have done. Recently I visited a packaging plant that used to think they were doing very well when they sealed 80 packages per minute; they are presently sealing between 200 and 300 packages per minute. The machines that they are using are capable of sealing 400 to 500 packages per minute and the only thing that is preventing them from doing this is an adhesive that will perform at this speed and does not cost 5 to 6 times more than that which they are now using. To top this off, I was just reading in a publication about a new machine that was being imported into this country, and the advertisement stated that the machine would make complete 3000 paper bags per minute. Just think of that—3000 bags per minute! I remember being on a test in Richmond, Va. before the war when a new machine was being installed that was expected to make 700 bags per minute. This machine was equipped with a Reeves drive, so before starting the machine we were a bit skeptical and set the Reeves for about 400 bags per minute. Everything seemed to work very well but the man who bought the machine wanted to see it perform at 700 bags per minute and he instructed the machine tender to rev it up. This was done and, gentlemen, the paste and glue flew in all directions. You never saw such a sight! The seam paste box was emptied in 10 seconds and glue was all over the shop. The bottom paste got so hot that instead of being pasty it became milky. Believe me, life was a bit unbearable for a while, but this did not stop us. We eventually developed adhesives that were satisfactory for this operation at the rate of 700 bags per minute and we will make adhesives to accommodate the 3000 bags per minute.

This is not an unusual problem for us to be faced with. As a matter of fact, in my mind this is what we have to look forward to in the very near future in all types of packaging and the manufacture of products from paper. We are endeavoring to gear our development work to meet these demands. Any help that we can get from the machine builders prior to the installation of new high-speed equipment and any advance notice that we can get from the paper manufacturers will be extremely helpful in aiding this cause. We do not seek this help and cooperation solely to shirk on our research work. We are installing and developing in our laboratories every type of apparatus for testing material that we can obtain or think of. We do seek the cooperation of the paper man and the machine man because we feel that the three of us have a mutual interest in seeing that the march of progress continues.

"In closing, gentlemen, I respectfully request that if you have an opportunity to be in on any new developments along the lines discussed, please give the adhesive manufacturers as much advance notice as you can so that this group, the papermakers, the machine builders, and the adhesive manufacturers, can continue to service the packaging industry in the same manner as we have in the past."

Lake Erie

The Lake Erie Papermaker's and Converter's Association met at the Hickory Grille, Cleveland, Ohio, on May 19.

The following officers were elected for the coming year:

Chairman—L. K. Burnett, Ohio Boxboard Co., Rittman, Ohio.
First Vice-Chairman—W. O. Manor, Consolidated Paper Co., Monroe, Mich.
Second Vice-Chairman—C. P. Spring, Chase Bag Co., Chagrin Falls, Ohio.

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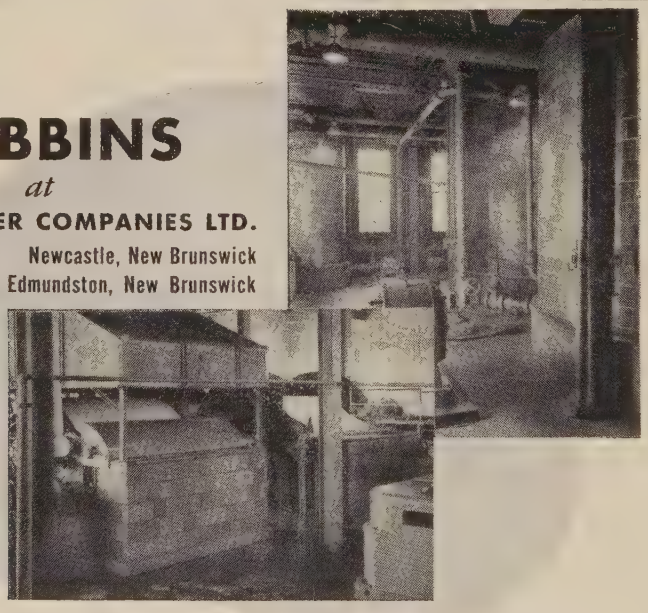

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The principal speaker was Joseph Steinbrunner, Asst. Factory Manager of the International Printing Ink Co., Chicago, Ill., who discussed "Printing Inks." Mr. Steinbrunner's remarks follow:

Printing Inks

"Basic research and product development in the ink industry have resulted in a great number of improvements over the years. Inks in bygone years were basically either oleoresinous or penetrating in nature and drying was achieved by oxidation, polymerization, penetration, and evaporation. From this simple beginning, printing ink manufacture has evolved into a precision industry, that is able to produce a product tailor-made for almost any printing process or end-point usage of the printed piece. Resin-solvent vehicles are used that will dry under proper conditions in split seconds.

"But these improvements would not be possible were it not for the tools, the testing equipment that plays the major role in evaluating theory on a practical basis. Our factories are equipped with printing presses of various types which are used by our laboratories to check the printed result of our experimentation under conditions analogous to those existing in the press rooms of our customers. We have single and two-color flat bed presses, single and multicolor offset presses, both web and sheet fed, a rotary typographic press which will print four colors on a web up to 1500 f.p.m., aniline equipment, and even a newspaper press of commercial speed. All of these presses have special equipment of our own design or selection that enables us to detect even minor advantages or disadvantages in one ink over another.

"No new development has any value unless it can be removed from the category of laboratory curiosities, be produced economically in quantity, and be controlled in quality. The most important factor and the one I would like to dwell on is the control that must be exercised to maintain high quality. Assuming that the ink produced on formula has been mixed and milled, a sample for testing is given to quality control. It is first checked carefully for shade by comparing it with an established standard prepared by the formulating chemist by simple drawdown. Then, by diluting the sample with from 20 to 50 times its own weight of opaque white, the chemist checks it for tinctorial strength and makes certain that the desired color value is present.

"While this is being done, manual examination of comparative viscosity has been made and a portion of the sample sent to the temperature and humidity controlled room housing our rheological instruments. We use two basic machines, the Interchemical Viscometer developed by the late Henry Green in our Research Laboratories and the Reed Inkometer developed by R. F. Reed of the Lithographic Technical Foundation. The I. C. Viscometer gives us the physical characteristics of the ink in dyne centimeters squared representing the tack, yield value and coefficient of thixotropic breakdown. These values taken at relatively low rates of shear must comply with the recorded values of the standard. The Inkometer, an empirical machine, operates at high rates of shear and correlates high-speed printing conditions. Between the two we have a complete picture of whether the ink will work like the standard.

"In the meantime bulking or specific gravity readings are taken and the dispersion examined on the fineness of grind gage. This latter device is a block of steel into which two wedge shaped grooves have been machined. The depth ranges from 1 mil to zero and after scraping the ink over these grooves, a source of light at the proper angle will allow the quality of the dispersion to be determined by visual examination. The groove is calibrated to allow accurate recording of the fineness of grind.

"Up to this point, we have merely made a production batch of ink, controlled to our specifications. Now we must assure ourselves that the ink will meet the customer's specifications. Comparative proofs are pulled on the exact stock on which the ink is to be printed. If the exact stock is unavailable, the proofer uses the closest approximation of the printers paper. These proofs are examined at frequent intervals to determine that the drying rate of the new lot of ink is equal to the standard. If the ink we have made will be used on printing machinery equipped with drying

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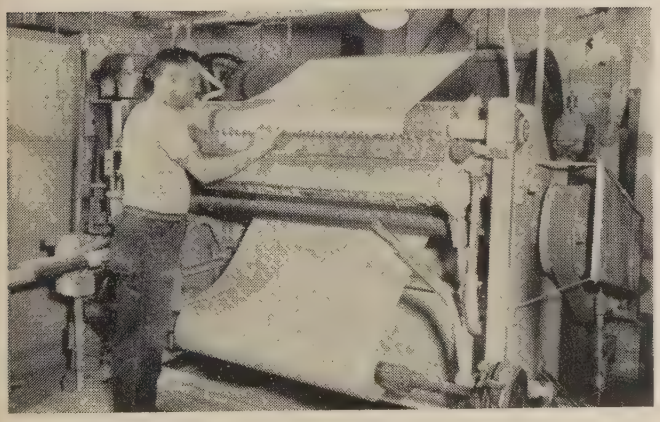

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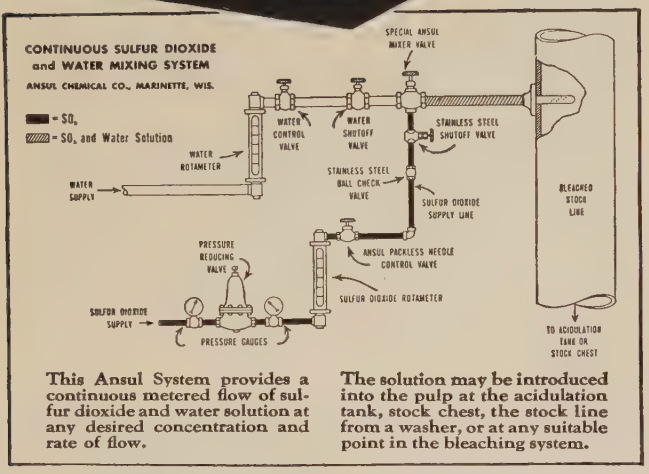

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4. Permits dissolving of organic lime compounds so they may be removed by washing.

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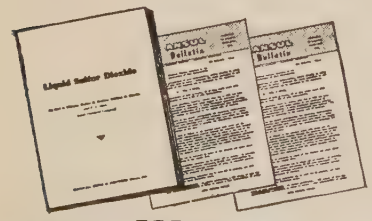

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devices, the drying time of the ink is checked on laboratory counterparts of the production equipment.

"After the proofs are dry, they are given to a laboratory physically removed from the production and control laboratories where chemists perform any other tests required. If the printed piece is to be used for a food wrapper, soap carton, lithographed metal can, cotton bag, or any other specific printed product, the ink must be approved by this testing laboratory. Apparatus in this laboratory has been carefully selected or designed as necessary to reproduce conditions that have been experienced in field results.

When the ink is found to have the characteristics required, it is approved for packaging. Again a sample is inspected for fineness of dispersion and this sample carefully labeled with formula number, batch number and date of manufacture, and filed for future reference.

"More than twenty years ago, it was recognized that a thorough and continuing study of the reciprocal relationships between paper and printing ink ("paper-ink reaction" for short) would be necessary to provide a basis for further research for the improvement of printing. IPI was among the earliest in this field of investigation. A paper titled, "Testing Printability of Paper with Ink," presented by F. A. Weymouth of IPI at the Annual Meeting of the Technical Association of the Pulp and Paper Industry at the Hotel Commodore in New York City in February, 1942, was probably the first successful attempt to correlate and standardize on methods of determining the relative printability of coated papers. The methods he described, by the way, have been adopted by a majority of the manufacturers of these papers. A second paper on the same subject was presented by Otto Berberich, L. F. Englehart, and Milton Zucker of the Research Laboratories to the same group at the same place six years later, based upon further studies by our laboratories in Chicago and New York. These papers are printed in the Paper Trade Journal of April 9, 1942 and August 12, 1948, respectively.

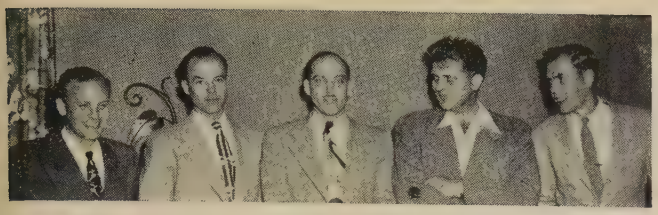
"The testing methods described by Weymouth have been found applicable to box board and other packaging material and have recently been adopted by some of the board mills as well as by producers of set up and folding cartons. Basically, there are two separate and distinct tests, one involving a visual observation comparing the ink receptivity of an unknown piece of paper against a paper of known printability by proofs pulled under controlled conditions and the second making comparisons of the known and unknown papers by proofing inks of progressively increased tack to determine the pick resistance. Please notice that in both instances, comparison against a known standard is required.

"Briefly, the printability test is performed on samples of the known and unknown which have been prepared by equalizing the caliper, and then affixing the two samples on backing piece to maintain relative position. The two pieces of board are then printed simultaneously on a precision proofing press, which has been inked with a minimum amount of ink. Because all of the contributing factors, such as ink film thickness, impression, speed of ink take off, etc. are exactly the same, the difference in ink take off is a true comparison of printability. An ink of low spectral reflectance will disclose such differences as wire marks, uneven surface, hard and soft spots, scabs, and pits.

"The second test, using inks of progressively increased viscosity, will reveal weakness of surface coating under conditions similar to those experienced in printing conditions. This test correlates the Dennison wax method of evaluating printing surface strengths but has an obvious advantage in that it does not affect thermoplastic binders frequently used in coating whereas hot wax may give erratic and misleading results on such coatings. The tack-graded inks developed by International Printing Ink, when judiciously used, will enable the board or paper manufacturer to predetermine the suitability of a particular stock for single, two color or four and five color printing, by comparing the unknown against a sample of known performance. This method must be strictly comparative rather than absolute since the film thickness of the ink, the temperature of the ink as varied by the time allowed for mechanical distribution, the humidity, the type of printing plate used and the speed of pulling the proof all influence the picking.

"While it must be recognized that the testing methods are empirical, experience has proved that paper mills using the laboratory proof press method of testing their product have shown marked improvement in quality. Their papers are known for superior printability.

"Because of increased interest in the production of printed material in gloss ink, another method of testing suitability of paper for proposed end-point usage has been developed. This test consists of proofing three standard inks of widely divergent composition. We use an orange ink of high pigment, low oil composition, a blue ink containing a small amount of pigment and a high percentage of oil and a red of high viscosity and medium oil content. Proofs of these inks can be observed visually and



L. to R.—T. Luey, P. Nethercut, W. Sprau, J. Harrison, and J. Dam

compared to a stock which is known to have the property of producing jobs of high gloss.

"It is not my intention to convey to you the impression that all printing faults can be corrected by the production of papers which will pass the printing tests described. We, of the printing ink industry, do not intend to shirk our responsibility in connection with the immediate production of a printing job.

"Too frequently the choice of paper is dictated without the necessary forethought that might have resulted in a superbly printed place. When an unsatisfactory paper is placed in production, the skill of a pressman can help to only a limited extent. The ink maker, having the greatest degree of flexibility, can usually adjust his product to further improve results, but the best ink or poor paper will not result in a printed piece of excellent appearance any more than poor ink will result in an excellent job on the finest paper. Good printing requires that the papermaker, inkmaker, and craftsmen combine their skills in order to produce satisfactorily printed copy without undue expense."

Kalamazoo Valley

The Kalamazoo Section held its annual Fun Day and election of officers at the Gull Lake Country Club on Thursday June 5. Over 150 people attended this meeting with nearly 100 playing golf.

John Hayes and Joel Sheppard tied for low gross with 77's to take top honors on the golf course. Other golf prizes were presented to Art Cole, Jack Hartung, Jack Brunner, Lee Mimms, Ralph Atkins, and Clifford Beebe.

First prize for the shuffle board play went to Robert Ronnigen and George Gerphiede Jr. with Rob Roy and Ray Burstadt taking second place. The Bogus money winner was Walter Shaw and R. J. Sues second.

The election of officers took place after dinner in the evening with three new officers chosen. Three new officers were necessary instead of the usual one because of the newly created office of recording secretary and because of the resignation of Fred Schucker as Treasurer. The following is the slate of officers chosen for the coming year:

Chairman—James J. Harrison, Michigan Carton Co., Battle Creek, Mich.
Vice-Chairman—John R. Dam, Allied Paper Co., Kalamazoo, Mich.
Recording Secretary—Philip E. Nethercut, Watervliet Pa. Co., Watervliet, Mich.
Corresponding Secretary—Thomas A. Luey, Sutherland Pa. Co., Kalamazoo, Mich.
Treasurer—Wm. J. Sprau, National Gypsum Co., Kalamazoo, Mich.

The committee in charge of the fun day this year included C. E. Mueller, Bulkley, Dunton Co., *Chairman*. Richard

L. to R.—C. B. Smith, R. Trelfa, W. Honey, and C. E. Mueller

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You'll want a copy if you are troubled with formation or dirt troubles—or if you are meeting the problem with relatively cumbersome and expensive methods.

The disintegration and dispersion of dirt is incidental to the total behavior of Bauer Pulp Refiners. So far as we know, no one ever bought these machines expressly for dirt diffusion, but rather to obtain the over-all advantages of better and more economical pulp processing and/or stock refining. *Anyway, ask us to send you the treatise.* No obligation, of course.

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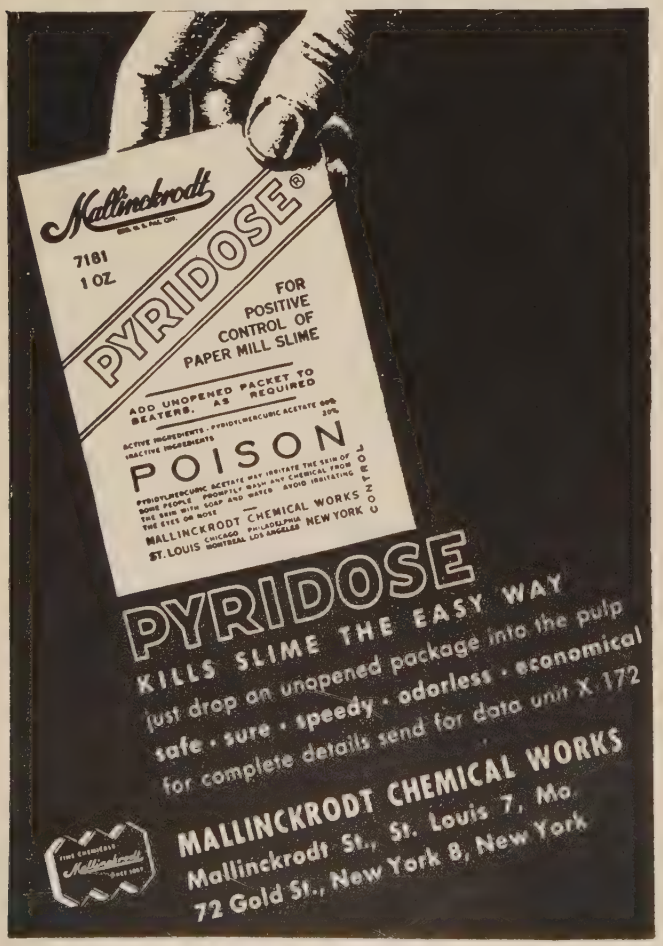
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Kalamazoo Valley Section

Empire State

Hudson Valley District

As previously reported, John C. Dean of the Socony-Vacuum Oil Co. presented a paper at the meeting of the Hudson Valley District of the Empire State Section, January 19, 1950 on "Waxes for Protective Packaging." Dr. Dean's remarks and the discussion of his paper follows:

Waxes for Protective Packaging

"The paper industry is consuming greater and greater quantities of petroleum waxes in their converting operations. This increased consumption is largely the result of developments in the packaging of foodstuffs. The waxes are used in bags, boxes, and cartons, in fact all types of paper and paperboard that are employed for food packaging. Articles packed in paper containers include frozen foods, precooked foods, and dehydrated foods—anything which is harmed by either loss or gain of moisture.

"The paper industry is by far the largest consumer of the petroleum waxes and is estimated to use 80% of the United States production. The petroleum industry is now producing between 800 and 900 million pounds per year of paraffin waxes and over 100,000,000 pounds per year of microcrystalline waxes.

"Petroleum waxes have been and will probably continue to be the cheapest moisture-vapor barrier available to the paper industry. Thus as consumption continues to increase production of petroleum waxes will expand at a comparable rate. Many synthetic resins are finding their way into paper converting applications, but it is doubtful if they will ever be used as extensively as petroleum waxes because of their inherently higher cost. Resins have a place in high quality packages, but where large volumes and low prices are necessary, it is doubtful if they will ever supplant petroleum.

"With increasing supplies of petroleum waxes and with a more demanding consuming public, there are definite trends toward higher quality. Paraffin waxes must be harder and more durable so that they will produce more lasting moisture-vapor barriers. In addition they should produce glossier films to enhance the customer appeal of the package. Today waxes are better than they were a year ago.

"Microcrystalline waxes are employed primarily as laminating agents for bonding two sheets of paper together. The wax then becomes an internal moisture-vapor barrier. Trends in the industry are toward microcrystalline waxes with greater adhesiveness and improved flexibility at low temperatures, the latter characteristic being demanded by the packagers of frozen foods.

"The petroleum industry is well aware of the more exacting demands of the paper converters and in many cases the wax refiners are modifying manufacturing techniques to produce finished products which more nearly satisfy the requirements. In some cases waxes of conventional quality are given additional refining to remove from them undesirable constituents. An example of this is an improved microcrystalline wax which no longer contains brittle and nonadhesive paraffin wax which detracts from its use as a laminating agent. The petroleum industry is also working closely with the paper converters on the use of resin additives to modify the basic properties of petroleum waxes. One of the most promising resins is polyethylene which has the ability to make waxes harder and tougher. It is being used today in fairly large quantities with paraffin wax and with blends of paraffin and microcrystalline waxes.

"One trouble experienced with paraffin waxes is that they oxidize rapidly and metals catalyze this reaction. In so doing they develop a rancid odor and have lower hardness and durability. Copper catalyzes the reaction more than iron, stainless steel not as much as copper but more than black iron. This trouble is being overcome by the use of antecedents or inhibitors. These are nontoxic and can be used in the paper usually impregnated with wax.

"Socony-Vacuum's Technical Service Laboratories at Brooklyn, N. Y. have been working with petroleum waxes for many years. During the course of this work they have developed new methods of evaluating the performance characteristics of the waxes as they relate to the paper industry. Recently they prepared a movie showing the methods used in the laboratory for producing experimental waxes and for controlling their quality. This movie is believed to be of interest to all paper converters since it shows the care which is exercised in producing what formerly were commodity materials of a byproduct nature. This picture will now be shown."

Discussion of Dr. Dean's Talk

(*Question*) What is the difference in physical composition of microcrystalline and paraffin waxes?

(*Answer*) I am not sure that we can say definitely but in general paraffin waxes are predominately straight chain paraffin components and the average length of chain is probably 24 carbon atoms. Microcrystalline waxes on the other hand appear to contain a substantial number of isoparaffins. The average microcrystalline wax probably has 36 carbon atoms in it. In other words, they are higher molecular weight and in addition are branched chain instead of being straight chain. Good quality lubricating oils are highly isoparaffinic. Microcrystalline waxes are chemically somewhere between the straight chain paraffin wax and lubricants which are more isoparaffinic.

(*Question*) I have two questions. (1) Do you suppose an oxidation inhibitor should be set by the buyer or user of the wax or is it incorporated in the nature of the wax?

(*Answer*) It should be applied by the consumer for the reason that it costs money to put it in and many people who handle wax do not have conditions severe enough to justify that added cost. We, for example, supply one that is a concentrate and recommend that it be used in amounts of 1%. It will raise your average cost about 0.2 cents per pound and that is more than a large consumer—a bread wrap manufacturer for example, who is working on a very narrow margin could pay, therefore we do not put it in. The basic inhibitor is dissolved in wax so that the mixing of it is relatively simple.

(*Question*) (2) Is the inhibitor you have for sale organic in nature?

(*Answer*) Oh yes, it is organic. It is soluble in wax to a sufficient degree.

(*Question*) Have you ever used any that is inorganic?

(*Answer*) No, we have not.

(*Question*) It would be acceptable in some applications?

(*Answer*) I cannot think of an inorganic inhibitor that would be soluble in wax and not have the problem of keeping it in dispersion. I think you are almost limited to an organic material.

(*Question*) Dr. Dean, is wax stable after it is applied to paper?

(*Answer*) I must make certain qualifications in answering that question, but I would say that by and large wax is stable, because in all the tests that we have run we get relatively little oxidation at temperature below 140–150°, and after it is applied to paper it is seldom exposed to temperature over 100–110°. However, there are certain types of butcher wrap that are made by impregnating with mixtures of petroleum, paraffin, and oil. We have had a number of complaints in the course of many years. I won't say a number but they are sporadic, one or two a year—from people making butcher wrap that claim rancidity develops in storage. There is no doubt it does, which means oxidation has taken place. In every instance the base paper involved has been bleached kraft. However, when we took samples of the wax, petroleum or oil, whichever it might be that had given the difficulty, and used that to impregnate an unbleached kraft, a bleached sulphite or an unbleached sulphite we had no development of rancidity. When we used the base paper, which was a bleached kraft, we got oxidation trouble. We suspect it was because of bleaching residues.

(*Question*) Did the use of antioxidants eliminate that odor on that particular sheet of bleached kraft?

(*Answer*) At that time the only antioxidants we had available were those that we were using with lubricating oils. But we were very reluctant to recommend them because we strongly suspected that they were toxic. There are a number of people who make butcher's wrap who always add an antioxidant to the impregnating material on the basis that, although they have to add 0.2 cents per pound of impregnant for all their paper, they do not have to take back carloads of finished paper which must be worked off at considerable loss. As far as I know those concerns have had no recurrence of the difficulty. It may be coincidental that they are doing better bleaching, or whatever it is to the paper to make it less catalytic. There is no doubt that petroleum oxidizes, but also that some catalytic action is the cause of it.

(*Question*) Dr. Dean, you said copper, zinc or steel might be bad, what about nickel?

(*Answer*) Stainless steel is not very bad. It is worse than carbon steel or black iron. It is not nearly as bad as copper. Copper-bearing metals such, as bronze and brass are in a class by themselves. Nickel, chromium, aluminum, stainless steel are fairly well down the line, but nickel is not as free from catalytic action as is iron.

(*Question*) Where is zinc in that series?

(*Answer*) It is along in that intermediate class. It is probably closer to iron and steel than it is to copper.

(*Question*) I believe the standard test for measuring the amount of wax in wax paper is carbon tetrachloride extraction, isn't it?

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(*Answer*) Actually I don't know that you would have to use carbon tetrachloride unless there was some rosin which was incorporated in the wax blend so that it would be difficultly soluble in petroleum solvents. In our laboratories we use petroleum solvents. We believe we remove all the wax. There is one advantage in using a paraffinic type solvent which is that you do not tend to dissolve so much of the rosin sizing which may throw your results off.

(*Question*) What solvents do you use?

(*Answer*) It is what you people probably know as petroleum ether.

(*Question*) Would that be well suited for paper with a very low wax content? Anything else better?

(*Answer*) With a low wax content the more anxious you should be to leave the rosin in the paper and not extract it, particularly if you are trying to identify the type of wax or to determine its characteristics. We use a Soxhlet extractor in our laboratories.

(*Question*) Then how do you separate the wax from the ether?

(*Answer*) Just by evaporation.

(*Question*) Have any trouble with the wax being carried off too?

(*Answer*) No, if you use a steam bath and air jet and don't heat it too long. However, you are perfectly correct. It is possible to vaporize some of the wax if it is overheated.

(*Question*) What would be the boiling point of 135° paraffin wax? Boiling point and flash point.

(*Answer*) I am not sure about the boiling point because you could not distill it without cracking unless you used a vacuum. The boiling range would be about 600 to 800°F. if you could distill it at ordinary pressure. The flash point will be in the neighborhood of 400°. However, if you get cracking of wax, which can take place, you will form gasoline. Cracking of heavy petroleum products to gasoline is conducted by heat and pressure. You make little ones out of big ones, so that it is possible in some cases to get a low flash point. If carefully run it should be around 400°F. Microcrystalline waxes will run higher; their flash points will be 500°–550°F., because they come from higher boiling petroleum fractions.

(*Question*) Dr. Dean, would you say polyethylene improves the durability and low-temperature flexibility of paraffin?

(*Answer*) It doesn't improve the low-temperature flexibility tremendously but it does make it more durable at low temperature. In order to get good low-temperature flexibility you must add a plasticizer. Microcrystalline wax is a typical plasticizer.

(*Question*) What are the optimum percentages of those two?

(*Answer*) To get fairly good low-temperature flexibility you must use 25 to 35% of microcrystalline wax. When you get in that range you are getting to the point where you are apt to have blocking trouble. For that reason it might be necessary to add 5 to 10% of polyethylene.

(*Question*) How do the prices of microcrystalline wax compare with that of paraffin?

(*Answer*) That depends obviously on the quantities you order, but they are somewhere in these ranges. Paraffin wax at perhaps 7 cents per pound; microcrystalline waxes, if you buy them in comparable quantities would be about 8 or 9 cents per pound, and polyethylene of course is quite expensive—that is up in the 40 to 50 cent range. You are making a high-priced wrap when you start using 5 to 10% polyethylene in the wax blend.

(*Question*) Does polyethylene help microcrystalline wax the same way as in the mixture?

(*Answer*) We do not see that it does enough by itself to justify its use. It is only when the microcrystalline wax is being used with paraffin. It is possible that some people are using polyethylene with microcrystalline wax and no paraffin. I don't personally know of any such use. It would seem to be throwing your money away.

(*Question*) Could you get or add greaseproofness to paraffin wax with polyethylene or would you have to use too much of it?

(*Answer*) I think you could, but you can only go so far with any petroleum wax with respect to greaseproofness. After all, waxes are soluble in oil or grease, and cellulose itself is not. If you have an open sheet of paper and you subject it to the action of grease, the grease is going to go between the fibers. If you fill those openings with wax, you will slow down the passage of grease. The degree of grease resistance you get is dependent upon the solubility of the wax in the grease. However, if you raise the melting point of the wax, you reduce its solubility in fat, which means you increase greaseproofness. For example by raising the melting point of wax from 125 to 165°F. you cut down the amount of grease that will pass through to $^1/_{40}$ of what it was. Using polyethylene will also reduce solubility so that you will enhance grease resistance but we are never going to get true greaseproofness. We are never going to get true greaseproofness with waxes for that implys that grease will never go through the sheet. You will get grease resistance. The only really greaseproof sheets are glassine, parchment, or cellophane where you have approached a continuous film of cellulose.

(*Question*) What are safe temperatures for storage and handling of liquid waxes?

(*Answer*) That too is a relative matter because you have so many factors involved. One of them is surface area. If you had a wax tank which covered a large area and only was 6 inches deep, you might say a safe temperature would be only 140°F. If you stood that tank on end so that you only exposed a very small surface area you might have a safe temperature range of 230–250°F. You also have the effect of metals which we talked about earlier. There is also the amount of agitation given the wax, so that it is very difficult to answer the question specifically. By and large for paraffin if you can hold your temperature down to 150°F. and have no copper present, you are fairly safe. For that reason you should never use superheated steam or high-pressure steam in heating the wax. It should be low-pressure steam or waste steam.

(*Question*) By low-pressure steam—how low?

(*Answer*) Approximately 3 to 5 psi., for waste steam. That will give only a temperature at point of contact of 215–220°F. Also you should use agitation to keep the wax moving beyond the hot point so as not to build up high local temperatures.

(*Question*) Do you get less oxidation at a specified high temperature with a low melt wax or a high melt wax? Or, isn't there any difference?

(*Answer*) In general high melting waxes are more stable than low melting waxes, even where only paraffin waxes are involved. When you get into microcrystalline waxes which also are high melting, you have two factors involved. One is the high melting point, but the more important is the fact that these waxes are seldom refined to a state where they are water-white in color. The colored bodies, contain oxidation inhibitors which are put there by nature. We know if microcrystalline waxes are refined to a very light color—almost water-white—they go rancid rapidly; much more so than those that haven't been given that degree of refining. That is one reason you will find very little white microcrystalline wax on the market. It costs a lot of money and it isn't very color stable.

(*Question*) What system would you recommend for keeping transfer lines to temperature?

(*Answer*) If you run a small pipe with low-pressure steam along the wax line and lag it with asbestos or some other material you can do a pretty good job. It is better to do that and keep the temperature down than to heat the wax to a high temperature and hope it doesn't solidify in the line. The lower you can keep the temperature of the wax at all times the better off you are.

(*Question*) What is the quickest laboratory method of checking the oxidation stability of your wax?

(*Answer*) The only one which we know is really meaningful is the method illustrated in the movie. Heat the wax to about 300°F. with oxygen and in 24 to 48 hours the end of the induction period will be reached and oxidation will start.

(*Question*) Do you check that by titration or peroxide number?

(*Answer*) Both peroxide and acid numbers are determined. You cannot rely entirely on either one alone because peroxides are constantly changing to acids. However, if only one determination is to be made, the acid number is the better of the two.

(*Question*) How about heating with a catalyst—say copper?

(*Answer*) It is difficult to reproduce results. Why? I don't know. We just use a glass tube with oxygen. It is an empirical test at best, and probably would be difficult to check from one laboratory to another.

(*Question*) Does the small amount of water you get in the bottom of a storage tank have any effect on stability?

(*Answer*) No. As far as we can tell water doesn't have much effect on oxidation. The greatest trouble you have with water is that when the wax is heated to over 212°F. the water tends to boil out of the wax and it starts splattering. As far as we know, it is the only harmful effect you are apt to get.

(*Question*) What is the future for the purchase of wax by specification or by properly described performance tests?

(*Answer*) I am going to answer that question by asking, what would you do if paper customers put more and more specifications on the paper that they purchase? The first thing would be that you would resist because it would cost you more money. On the other hand if the entire trade insisted on it, you would do it. We have the same situation with wax. Obviously, the more we can turn out by standard methods the better we like it because we keep our costs down. On the other hand competition and customers may force the industry to more specifications than exist today. As a matter of fact, some people buy less on melting point than they do on the hardness at 100°. It is going one step further along the line. There is a committee operating in TAPPI and also in ASTM to develop various test methods. We ourselves feel certain of these methods being meaningful. Some we are a little skeptical about. I would say there is a trend away from commodity type purchasing to insistence on more binding specifications. Within our own company our manufacturing specifications today are a great deal stricter than they used to be.

(*Question*) What is significance of tensile strength in waxes to, let us say, elasticity?

(*Answer*) Tensile strength is somewhat a measure of durability, toughness and cohesiveness. Flexibility has no definite relationship to tensile strength. For example, you can have a wax of low tensile strength and which is very flexible. Another difficulty is found in the measure of tensile strength. Conventional procedures give the wax briquet a rather sharp break, and you can perform some rather peculiar gyrations with wax blends. If you add 3–5% microcrystalline wax to paraffin you can increase the tensile strength by the methods normally used by as much as 50%. However, when you add 10% microcrystalline wax the curve takes a drop and you get below the original tensile strength of paraffin. Finally if you add 20–25% the tensile strength goes back up again. I would believe the 10% blend, even though it has a lower tensile strength would be a tougher, more durable wax than the one containing only 5% microcrystalline wax.

(*Question*) What customers are using the microcrystalline wax, polyethylene combination—what are they making?

(*Answer*) Blends of microcrystalline wax, paraffin wax and polyethylene are used on frozen food wrap, locker paper, and even bread wrap among other papers.

(*Question*) What type of wax is used in wax size?

(*Answer*) Most of it is paraffin type wax although microcrystalline waxes and even petrolatums are employed.

(*Question*) How are they emulsified?

(*Answer*) A wax emulsion is not an emulsion in the physical chemist's sense of the word. It is more of a dispersion because it is a solid in a liquid rather than a liquid in a liquid. In general there are two types of wax emulsions. One type is emulsified with a soap, but it is not regarded with very much favor with the paper industry since such an emulsion breaks quickly if hard water conditions are encountered or if pH is much below 7.0. This is particularly important in beater sizing. The other type of emulsion is made with proprietary emulsifiers which are in themselves resistant to hard water and to acids. Therefore, the emulsion is resistant. The natures of the proprietary compounds are rather closely guarded secrets of the manufacturers of wax emulsions.

(*Question*) Is there any strength loss using wax size?

(*Answer*) Yes. If you size in the beater you must admit there is some loss.

(*Question*) Does it act as a lubricant?

(*Answer*) Probably that is part of the answer. Treatment at a

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size-press has less effect because formation has already taken place. Wax emulsions have a softening effect that is advantageous for such things as wall hangings, tablet paper, and the like. They also tend to reduce curl. There are some places where the loss of strength is advantageous. I understand that people making milk bottle caps with lift tabs frequently put wax in the inner ply of that sheet deliberately to soften that ply so that it is very easy to pry up the tab.

(*Question*) When you speak of durability of wax on a waxed sheet of paper exactly what do you mean?

(*Answer*) I mean resistance to marring of the wax film in handling.

(*Question*) Would it be correlated with tensile strength?

(*Answer*) It would be correlated to an extent but there are other factors.

(*Question*) These antioxidants you were talking about—you say you supply them in a wax solution?

(*Answer*) Yes, because the active ingredient we are using is very difficult to make soluble and it would be rather a problem for a mill to put it into the wax directly.

(*Question*) What is the physical connection between polyethylene and paraffin? There is no chemical combination is there?

(*Answer*) Probably if you look at a mixture under a microscope you would find a colloidal dispersion. By and large anything soluble in wax will have very little effect in modifying its properties. You are dealing in colloid chemistry.

(*Question*) How would certain resins affect it?

(*Answer*) Such as.

(*Question*) Well, the vinyls?

(*Answer*) They are quite insoluble. Take the commonest resin—ordinary rosin. You can put rosin in wax. In fact it is used occasionally with microcrystalline wax to enhance its binding properties. The difficulty is that the resin tends to oxidize, and when it oxidizes it becomes insoluble. You end up with a gummy deposit in your wax tank. Anything that is highly unsaturated would probably do the same thing. You can take other resinous materials which are soluble in wax and you will find they have little effect in varying the properties unless you use large quantities. Here you are dealing with a one phase system and are getting away from a colloidal dispersion. You don't get the effect of the resin in this case.

(*Question*) Is it possible to make an emulsion with paraffin and precipitate a continuous film of wax on paper?

(*Answer*) The only way you could do it is to precipitate the emulsion and subject it to heat so that the individual wax particles are fused into a continuous film. In the emulsion they are separated by the protective colloid, after they are precipitated the boundaries between the wax particles probably still exist. It would require laying down quite a thick film of wax which would be quite difficult. I don't know that anybody has done it on paper. You can do it on wood or on substance that is not so fibrous as paper.

(*Question*) What has been your experience about the flexibility remaining constant after microcrystalline wax has been applied to greaseproof paper? Does temperature or anything else affect that?

(*Answer*) You might affect the flexibility of your finished sheet if you store your paper at a slightly elevated temperature where the wax would tend to soften and become absorbed into the board. Then you would not have a continuous film of wax which might detract from flexibility. The fact that you have two sheets with no real lubricant between them might detract from flexibility. I would think it would be more the effect of the paper than of the wax.

(*Question*) We tested the paper for flexibility before we stored it. This board was laminated with parchment. We had considerable difficulty with that board.

(*Answer*) Was the paperboard humidified before laminating?

(*Question*) No, we laminated with wax.

(*Answer*) Let me explain. If you take parchment and expose it to a dry atmosphere it will lose moisture and become less flexible. Is that correct?

(*Question*) Yes.

(*Answer*) It is possible that in the laminating operation you have subjected the paper or the parchment to an elevated temperature and have dehydrated it. That might be enough to make it so stiff that you would have this difficulty. I would think one way to overcome the difficulty would be to humidify slightly as you are winding it. There is another factor that is involved in laminating glassine to glassine. A plasticizer is often added to the wax so that the wax does not detract from the basic flexibility or softness of the glassine, which itself is usually plasticized internally. Viscous oils of one type or another are used as plasticizers. They, of course, have no effect on the paper, but are used to make sure the wax is as flexible as the paper.

Meeting of May 18

The Hudson Valley District met at Milfrank's Restaurant, near Glens Falls, N. Y. on May 18.

The following officers were elected:

Chairman—Robert Moynihan.
Vice-Chairman—Hugh H. Lavery, International Paper Co., Palmer, N. Y.
Secretary-Treasurer—Grant Cole.
C. N. Hagar, Jr. of the BFD Div., Diamond Match Co., Plattsburgh, N. Y. Past-Chairman was elected to the *Executive Committee.*

The speaker was Walter Mikelson of the General Electric Co., Schenectady, N. Y. who discussed "Isotopes in Industry." Mr. Mikelson pointed out that radioactivity is as old as the sun, which is an example. Its oldest uses by man, he said, are x-rays and radium which were discovered by their effects on photographic plates and their ability to cause fluorescence in certain materials.

The speaker said that in the near future the use of radium may be replaced by less expensive materials of more controllable intensities. In describing types of radioactivity he termed alpha rays as having slow moving particles and low-depth penetration, beta rays as being faster and more penetrating, and gamma as having no mass, high velocity, and high penetration. All are dangerous to man if not properly used. On this point he said that if the instructions regarding safety that come with instruments having radioactive components are followed, the danger is no greater than from the luminous crystal of a watch.

Examples of the uses developed for these instruments included measuring of temperature, vacuum, pressure, weight and thickness; determining the depth of drilling levels, the performance of lubricants, the evaluation of sulphur and phosphorus in steel, and measuring the depth of the seal on a drill hole.

A film on logging, by Finch Pruyn and Co., by the use of a Wissen cable was shown.

Delaware Valley

The Delaware Valley Section met at the Engineers Club, Philadelphia, Pa. on May 25. The meeting was called to order by Chairman John P. Weidner of the Container Corp. of America, Manayunk, Pa. He thanked the members for their part in the year's financial success which followed the good response to requests for dues payments. Chairman Weidner stated that he was proud of the job done by his staff of officers for the year coming to a close and that he was turning over the reins of the Section to Gus Erspamer, who had done so well as Program Chairman for the year 1949–1950. Dr. Erspamer thanked John Weidner for the introduction and stated that his hope was to do as good a job in the coming year as Chairman Weidner had done in his two years as Chairman. The other officers who are to serve during the coming year were introduced to those present. These are: Arthur J. Haug, *First Vice-Chairman;* Harold Brill, *Second Vice-Chairman;* Frank Lovegren, *Treasurer;* and Emil Padavic, *Secretary.* Acknowledgments were made, specifically, to John Macadam, who arranged the Maintenance Panel Meeting program, and to Jack Harper, who has devoted much time and effort in managing the name badges. The badges have been very helpful in promoting better acquaintance between members.

John P. Weidner, retiring chairman of the Delaware Valley Section of TAPPI

Dr. Erspamer presented Moderator Harry C. Merritt, Downingtown Mfg. Co., Downingtown, Pa., who took charge of the evening's Panel Discussion.

The Panel Members included:

C. M. Connor, W. C. Hamilton and Sons, Miquon, Pa. George E. Ellis, Jr., Scott Paper Co., Chester, Pa. C. F. King, Westinghouse Electric Corp., Philadelphia, Pa. N. B. Rohrbaugh, P. H. Glatfelter Co., Spring Grove, Pa. Karl R. Wolf, Scott Paper Co., Chester, Pa.

A questionnaire, "I would suggest the following subject for discussion by the Panel on Paper Mill Maintenance at the May 25 meeting," was distributed to those in attendance at the April 27 meeting. The responses were used as the basis for the evening's discussion. The moderator read the questions, and after each question referred it to the various panel members who voiced their opinions, experiences, or other comments concerning the question under consideration. The moderator and members of the audience also participated in the discussion.

1. What can the paper-machine builder do to aid mill maintenance?

Mr. Ellis: Design engineers should give full consideration to the fact that their machine will need repairs and provide for it. For example, the design should allow bearings to be pulled with relative ease and a minimum of time loss; types of material should be considered; and driers should be equipped with dippers or siphons which can be inspected and replaced readily. Builders should caution the owners concerning possible results if the owners' suggested designs are used.

Mr. Wolf: A builder installs a machine on the floor of his shop, whereas, oftentimes a machine installed in the mill has been designed so that disassembly is impossible without resort to use of contortionists.

Mr. Rohrbaugh: How is the machinery builder to find out? If a builder would send service men, at no charge to the buyer, they could check on the proper design, and also check on what is wrong with installed machinery. In this way the builder would strengthen his position in his field.

Mr. Connor: Buyers don't always give all the data needed by the builder. Too often buyers fail to avail themselves of services which the builder offers.

Mr. Merritt: In defense of the machine builders, designing draftsmen are just that and not papermakers. Sometimes an idea from one customer is included in a standard design and then the builder finds that the next customer wants no part of the idea. This is one of the things which makes it so difficult to establish a universally satisfactory design on a machine.

Mr. Ellis: Maintenance is interested in the least amount of down time. To attain this an exchange of ideas is required between staffs of customer and builder. An example of this is in the selection of a type of pump for a particular job.

2. What piece or pieces of equipment or section of machinery requires greatest attention, and are there any suggestions which would help the manufacturer to design a more satisfactory unit? What part of the mill requires the most maintenance?

Mr. Ellis: A paper mill is so integrated that it is hard to divorce sections. I would suggest drier joints, so important to drying; there can be no slipping chain drives; and on high-speed-machines rolls must be dynamically balanced. All parts are related so that maintenance is one over-all problem.

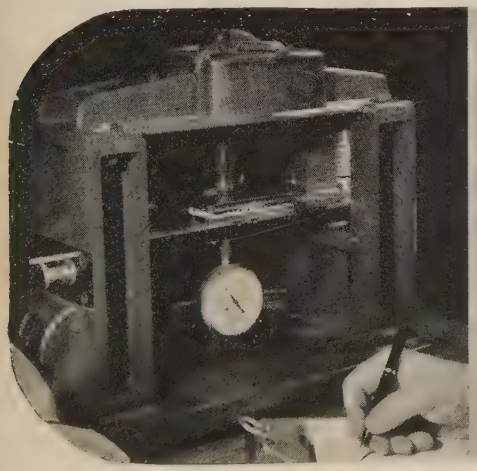
Mr. Wolf: I'll go along with Mr. Ellis. On a Yankee drier, maintenance borders on loving-kindness. It is inspected at every opportunity and grinding and polishing takes place when necessary. This creping drier is the most delicate part and gets the most attention.

Mr. Rohrbaugh: There are two critical parts. The first is the calender stack where the rolls must have a good, ground surface and have the proper crown. The second is the wet end where the sheet is formed. This is particularly true of the fourdrinier machine, especially, the inlet which does not require much maintenance but must be right. Its operation or design is not up to maintenance but the responsibility falls back on them when something goes wrong.

Mr. Merritt: Each mill presents a different set of conditions. By recording the amount of maintenance required, the most troublesome part will become evident.

3. What is the relative importance as applied to special equipment, such as, electric drives, of: (a) Preventive maintenance on plant level? (b) Outside services? (c) Replacements?

Mr. King: Good electrical maintenance keeps ahead of the papermaker so that there is no loss of production due to electrical failures. Whether the maintenance is done by plant personnel or outside help depends on the experience, ability, and



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knowledge of the inside help. When I think of paper-mill electrical equipment, I think, principally, of sectional paper-machine drives. These require a broader knowledge of electrical fundamentals for satisfactory operation and maintenance than do other drives. Our general practice, which may be extended if necessary, is to keep in close touch with the electrical installation force to render advice when the installation is being made. Following installation we make checks and adjustments, put the drive into service and instruct the plant personnel. This program, in the large majority of cases, results in the mill electrical organization being able to take over. Renewal parts should be purchased from the manufacturer of the apparatus with which the parts are associated. Usually, the mill electrical force is capable of installing such parts.

Mr. Wolf: In order of importance: (1) preventive maintenance, (2) replacements, (3) outside services. We make every effort to get equipment for which service is available locally.

Mr. Rohrbaugh: Preventive maintenance in the plant must depend on outside services until the inside help gets sufficient experience to handle it. The distance from outside services is a factor. The greater the distance, the greater the importance of plant level maintenance.

4. Stock pump maintenance, how often?

Mr. Connor: I presume the question concerns scheduling. With good record keeping and barring breakdown, the normal lubrication and packing attention required becomes a matter of mill experience.

Mr. Rohrbaugh: A plunger pump requires more attention than a centrifugal pump. At P. H. Glatfelter Co., a running check is made, externally, once a week and the pump is opened once a year.

Mr. Joseph Armstrong (Scott Paper Co.): I don't know that I can say any more than has been said. We make rigid inspections and carry spare parts on hand. A wire (from pulp bales) condition may allow indefinite operation time or it may cause trouble in 24 hours.

5. What is your choice of oil for paper-machine lubrication?

Mr. Connor: It depends on the recommendations of the manufacturer of the equipment or the manufacturer of the auxiliary equipment, such as bearings. For example, tests were made to determine the best lubricant for a supercalender operating at high pressures and high temperatures. I would suggest the services of a lubrication engineer but would be guided by the machine builder's experience.

Mr. Wolf: Oftentimes manufacturing equipment is not used as it was designed to be used. In such a case the lubrication engineer is called on in an attempt to solve the problem. The lubrication oil supplier will be able to recommend the proper lubricant, if he has a proper knowledge of operating conditions.

Mr. King: For sleeve-bearing motors in paper mills, a well refined, pure petroleum oil, free from acid, sediment, dirt or other foreign material, having a viscosity of from 180–220 seconds SAE at 100°F. and having a maximum pour point of 35°F. is, generally, suitable.

6. What is the proper routine procedure for cleaning of suction press rolls to keep them in top operating condition?

Mr. Ellis: This depends on the mill. In some mills the holes will clog and in others this is no problem. At Scott Paper Co., suction press rolls are pulled according to a regular maintenance schedule and each roll gets a major overhaul once a year. If the vacuum on the roll is watched and records kept, it will be shown when the lips need attention and whether the cleansing program is adequate.

Mr. Rohrbaugh: At P. H. Glatfelter Co., we have the problem of holes plugging on both suction press and suction couch as our stocks contain plugging materials. So far, the manual methods of drilling and punching are being used. From a mechanical standpoint, the rolls are opened twice a year to determine the condition of the inside of the roll.

Mr. Connor: The most modern method is the use of high (1000 p.s.i.) pressure water applied while operating the roll. This is more successful than drilling where there is a danger of breaking off drills. Following the old method, the roll is boiled out with a hot alkaline solution, where the temperature is maintained a 150–160° for four hours. Then eight men on a side drill for four hours. Using this method the machine was down for eight to ten hours every 90 days. Using the Hydrosilica pump every time the felt is changed there has been no need for drilling since 1946.

7. How do you control oil leaks on vertical stack driers?

Mr. Merritt: The problem is the same on a leaking bearing on any drier.

Ray E. Stearns (Empire Box Corp., Stroudsburg, Pa.): My experience is that it is practically impossible to keep an oil bearing from leaking. With a proper grease for the temperatures encountered, the oil can be eliminated.

Mr. Merritt: It comes down to getting a bearing that doesn't leak.

8. What is the value of a good steam joint?

Mr. Merritt: This is a sore subject.

Mr. Ellis: A steam joint presents a problem with salts in the steam, carbon disks, etc. The answer is to keep on top of their maintenance, replace them religiously and as soon as possible when failure occurs.

Mr. Rohrbaugh: A good steam joint results in a reduction of the electric horsepower to drive the drier section.

Mr. Connor: It is possible to reduce the amount of oil in condensate going back to the boilers with good joints.

Paul E. Rogers (Scott Paper Co., Chester, Pa.): I don't know what thought is behind the question but assume that it is horsepower. Some joints are designed so that the steam pressure is directed so as to tighten the joint.

9. How often should motors be cleaned and repainted?

Mr. King: I don't believe an answer to this question can be given which would apply to motors in all paper mills. Operating conditions vary widely, for examples: temperature of windings, amount of moisture and chemicals in the ventilating air. Deterioration or thermal aging of the varnish on windings of motors renders the basic insulating materials vulnerable to failure in the presence of water, oil, dirt, and other contaminants. High operating temperatures accelerate thermal aging. One of the large manufacturers of paint has a slogan, "Save the Surface and You Save All." This might well be applied to motor windings. It is good practice to megger the windings of motors at regular intervals. The insulation resistance at 75°C. should approximate one megohm for each 1000 rated volts of the motor. This should, approximately, double for each 10°C. reduction in motor-winding temperature. When the insulation resistance continues to drop between tests or when there is a more than average drop between tests, it is time for the windings to be cleaned with one of the numerous petroleum distillates available for this purpose and revarnished. In the paper industry, as in other industries, "Cleanliness of the Electrical Equipment is the Big Brother of Continuous Production."

C. J. Shafer (Empire Box Corp., Stroudsburg, Pa.): Using a drying varnish such as Glyptol isn't there a chance of driving moisture into the winding? How can a motor be dried without special equipment?

Mr. King: The motor should be dry before varnishing. A high megohm reading indicates dryness.

Mr. Connor: In most mills it is possible to rig up unit heaters, etc. High-pressure steam may be used in an emergency to dry to a safe point and then the motor be allowed to dry in operation, fairly successfully, where no prolonged drying is advisable.

Mr. Shafer: How about a low-voltage current across the line?

Mr. King: That is all right if care is taken to be sure the current doesn't overheat the windings.

10. How is your instrument group handled?

Mr. Wolf: At Scott Paper Co., the Instrument Department is in the Engineering Division. We have approximately eight men who have been trained on the job and they have done an excellent job. Their work is divided up into sections and they handle all instruments except the electrical. Their training is augmented by training periods at instrument manufacturers' plants and schools. The department has been operated with good continuity but is not perfect, yet. New men must be trained and it takes six months to a year for the men to be able to find their way around the mill.

Mr. Rohrbaugh: We have three men at P. H. Glatfelter Co. They were hand-picked and started under the electrical foreman. Recently, they have been cut loose from this foreman but are under his supervision. Our instrument men are trained at the plant. We also have these men receive training by attending the courses offered by the instrument manufacturers.

H. H. Street (National Vulcanized Fibre Co., Yorklyn, Del.): How many instruments does one man maintain?

Mr. Rogers: At Scott Paper Co. one man takes care of over a hundred instruments. We expect to get this down to about sixty instruments per man.

Mr. Merritt: Does each man take care of all types or are they specialized?

Mr. Ellis: They are not specialized.

Mr. Wolf: We have a repair crew of three who spend full time on repairs. The other five are divided as to section responsibilities.

J. Howard Wright (W. C. Hamilton and Sons, Miquon, Pa.): How are paper-testing instruments maintained?

Mr. Wolf: These are under the jurisdiction of the testing laboratory. No differentiation is made between testing and control instruments.

Arthur J. Haug (Scott Paper Co., Chester, Pa.): The Instrument Department has always serviced Testing Department instruments. They are naturals for this job.

11. How are your paper-machine preventive maintenance shut-downs scheduled? What is the best method?

Mr. Merritt: Regular clothing changes and week-end shutdown should, properly, be integrated into this schedule.

Mr. Rohrbaugh: At P. H. Glatfelter Co. preventive mainte-

nance is scheduled according to clothing changes, especially, wire changes. After three weeks of wire life, the maintenance department tells the operating department how much time will be required. They are then allowed so much time by the operating department and jobs are scheduled accordingly.

Dr. Haug: How much time?

Mr. Rohrbaugh: Six to eight hours on some wire changes, none on others. This shutdown time is from paper off the reel to paper on the reel following the shutdown.

Mr. Wolf: How long do felts last?

Mr. Rohrbaugh: Felt changes are made every four weeks on first presses and eight weeks on second presses.

Mr. Wolf: With us, arrangements are scheduled with other clothing rather than with wires.

Mr. Ellis: The question of how often and when depends on many things. Six months for some items and yearly for others narrows it down. We try to build the job around the maximum allowable downtime. The economics of downtime costs must be considered with caution. Such considerations of flexibility and interchangeability add up to length of time the machine is down.

12. What system of "preventive repair" can be suggested as better economy?

Mr. Connor: Mr. Ellis touched on this. By speeding up maintenance it is possible to spend money without knowing it in loss of operating time. Money spent in prolonged maintenance may be returned many times over. A good job of cleaning results in a cleaner product. Another gain is that of less damage from mechanical failure due to a hurried repair job. A shower pipe through a wire or a bolt through a press as compared with highest possible felt and wire life all add up.

Mr. Wright: Is there an advantage in having specialized mechanics in the machine room or in having men assigned to a particular machine or machines?

Mr. Wolf: At Scott Paper Co. each machine has a man who is responsible for bolting couplings, wire guides, oiling, etc. He checks on the oiler but does not relieve him of his primary responsibility. In the service shop there are various experts. One man may be an expert on wire guides, for example. These men are chosen for their definite mechanical aptitudes.

Mr. Rohrbaugh: At P. H. Glatfelter Co. there is one man on each tour who services all the machines. He is one of the manufacturing personnel and directly responsible to the operating superintendent.

Mr. Ellis: At Scott there is a man on each tour besides the specialists.

Mr. Merritt: What are your oiling personnel schedules?

Mr. Rohrbaugh: One man on each machine does the oiling and one man on the day shift checks all oiling.

Mr. Ellis: We have an oiler around the clock. The paper-machine mechanic is responsible for the close oiling and greasing while running. He checks on maintenance, too. We get good coverage by this means. Also, the mill engineer, who is on each of the three shifts, cooperates with the oiler.

Mr. Wolf: The electricians oil and grease their motors.

Glen T. Renegar (Container Corporation of American, Manayunk, Pa.): What is the cost of maintenance per ton of product? How much is allowed in the budget for the entire mechanical department?

Mr. Ellis: Our costs are figures on the cost per case and we are kept on the target by this cost. We keep a forty hour week, hold clothing changes to a minimum, and keep machines running to do this.

Mr. Renegar: Is maintenance help cost kept separate?

Mr. Ellis: We have a maintenance pool. Machine mechanics are in this pool, also, riggers, etc. Not including converting maintenance men we have about 140 and with the electricians about 170 men. We believe it pays dividends.

Mr. King: Who furnishes oiling specifications and schedules to maintenance men?

Mr. Ellis: This is done on regular schedules with routine checks.

13. What type of work-order system works best? What is needed on this type of form?

Mr. Connor: They should be the simplest which can be devised to meet the conditions. They should provide for man-hour distribution of expense. They should, also, provide for a report as to whether the job is completed or needs further attention.

Mr. Rohrbaugh: Our system works, anybody may request a job. This is approved or rejected by the department head as a first screening. The department head may have to do some fast talking, at times, to explain why a job is not done, but that is part of his job. The equipment number assigned by the plant must appear on the request. Also, an accurate description of the work to be done must be on the request. If not, the request is returned to the department head. A second copy of the work order is given to the foreman in charge of the job and this is returned to the Engineering Department where costs of labor and materials are figured. Each mechanic lists the time spent on each job by job number.

Mr. Ellis: In our work-order system, each department head screens the work requests. The mill is divided up into three sections. For example, the Paper Division Manager routes a work request to the Job Planner who investigates the job before giving it a job number. Upon completion, Cost Accounting gives a detailed cost of the job. The cost of maintaining any section is readily available.

14. Who should be responsible for proper start-up of equipment after a breakdown repair?

Mr. Connor: The operating superintendent should be responsible on production equipment. Where a maintenance man is assigned to a specific machine it is easier to place this responsibility. It is more difficult if the maintenance foreman is responsible. The production superintendent is responsible for any loss of production, so, he had better be sure that the machine is ready to go.

Mr. Wolf: There is no argument at Scott. The man running the machine is responsible but the maintenance shop responsibility comes back home if anything goes wrong.

Mr. Merritt: Who tells who it is ready to start?

Mr. Wolf: The job responsibility is listed on the job check list. Where various sections are being worked on together it seldom happens that they come out even. The Division Manager is responsible, but the Planner is normally on the floor and helps to coordinate and arrange for starting-up.

15. What is the proper sequence for a paper machine shutdown to avoid fiber loss, stream pollution, etc.?

Mr. Connor: It depends on the conditions. With slow running machines it is possible to do much to lower losses. Not just pull switches and open clutches. On machines running up to 400 f.p.m. it is possible to take stock off the presses by slowing down on the wire. In order to have no stream pollution there is not going to be any reduction of the load on recovery equipment.

Mr. Wolf: In running machines at speeds of 1700–1800 f.p.m. the problem in shutting down is to keep from flooding the place. We run down the storage chest at a definite time before the scheduled shutdown, then run down the machine chest as low as possible.

Dr. Haug: Our Waco filter doesn't handle mill shutdown waters. The white water is carried away in such a manner as not to exceed pump capacities. With a single machine shutdown it is only a matter of one or two chests, whereas, on a mill-wide shutdown, the problem is greatly accentuated.

Mr. Connor: As to sequence, the savealls should be shut down last in order to lighten the load on other recovery equipment as much as possible.

16. "Saveall Efficiency"—What is average efficiency for fibers?; for fillers? What are common errors made in using them and can efficiency be improved?

There were no comments on this question.

H. L. Berg (Bulkley Dunton Pulp Co., New York, N. Y.): In regards to suction roll cleaning, I tried to interest some roll manufacturers in a Finnish patent some years ago. The rolls are cleaned with a jet of water at 150 p.s.i. which oscillates over the turning roll. Four rolls may be cleaned in eight hours. Once a month is the average. Practically, all Scandinavian mills are using the method.

Mr. Wright: How can moisture-test samples best be taken to determine the moisture content of the sheet leaving the suction press roll on Yankee drier paper machines?

Mr. Rogers: On a pickup machine we use an air hose blast to blow the sample into the moisture test can. We get about 4% accuracy between individual determinations. Air at 25 p.s.i. may be used.

Dr. Erspamer in commenting on the program for the coming season, which is being developed, stated that the types of programs and the program material used would depend upon the wishes of the members. Members and potential members are invited to submit their comments. There was only about a 25% response to the evening's questionnaire regarding suggested program subjects. Dr. Erspamer requested that everyone who had not submitted an answer drop a line to Dr. Haug concerning the types of programs which would interest each individual, personally.

Dr. Erspamer adjourned the meeting with a call for a standing vote of thanks to the Moderator and Panel Members by the 75 members and guests present.

FRANK J. LOVEGREN, *Secretary*

FIFTH ENGINEERING CONFERENCE

Cincinnati, Ohio — Oct. 2–5, 1950

Control of Sewer Losses

JOHN G. DOBSON

THE continuing and ever-increasing parade of new statutes and regulations, both State and Federal, covering stream pollution, is making every mill more and more conscious of the material which is going down the drain.

There has been much loose talk, in discussions of industrial waste and pollution control, asserting that by proper design of treatment plants, material could be recovered which would make industrial waste treatment a profitable venture, rather than a liability to be carried by the other manufacturing processes. Experience has proved that except in unimportant exceptions waste treatment methods are usually not self-supporting.

However, the savings, due to changes in processes and improvements in housekeeping in our industries, have often been successful in paying their way and showing a profit, as well as decreasing the stream pollution load.

The various possible process changes in pulp and papermaking operations which may be used to decrease stream pollution and at the same time show savings to the industry, such as the closing up of white water systems and the use of spent cooking liquor evaporators and furnaces, have been already discussed so completely that anything which the author might say about that type of change, at this time, would be duplicating material already well known.

However, assuming that those process changes have been made and the mill has been thoroughly modernized, there is still one further step which can be taken in improvement of plant operation. This step seems to have been little appreciated generally. By improving the general housekeeping, by better care of operations, and by systematic general maintenance of the plant, substantial and sometimes surprising savings can be made in chemical cost. The savings are in preventing chemical losses to the sewer; not in trying to recover those chemicals once they have reached the sewer. For instance, in one modern integrated mill producing kraft, the savings in salt cake alone, from the beginning of a program similar to that to be discussed, resulted in decrease in chemical usage of nearly 100 pounds of salt cake per ton of pulp, with a net saving in salt cake cost alone of about $90,000 per year.

The sewer losses which occur, both in chemical and in fiber due to such minor operating irregularities as a tank running over, or foaming, or due to such minor mechanical causes as a pump packing gland leaking, or a small hole in a saveall screen, are seldom appreciated either by the management or by the man responsible for preventing them.

If the system for control of sewer losses is to be efficient, it must perform two separate and distinct functions. It must first, give an accurate estimate of the actual monetary loss to the plant because of the poor housekeeping that may exist in the plant, so that the management can see the justification for improvement in housekeeping and improvement in care of the entire plant; and second, the survey must place in the hands of the men actually maintaining and operating the equipment indications as to when the sewer losses are occurring and how much they amount to in dollars and cents. Placing of such information in the hands of the operator will often develop his interest to the point that he will be more careful in handling his operations. Also, by having comparative records of the operation, by the operators on different shifts, it is often possible to develop a spirited friendly competition to see which shift can do the best job.

A third and less important function which can be served by a continuous survey of sewer losses is the establishing of permanent records of the actual quantities of polluting materials which are being discharged to the stream. As regulatory authorities become more strict in their supervision of material discharged to the stream, and as some of them become more legalistic in their viewpoints, it seems probable that a daily record of actual polluting materials discharged to the stream may be of valuable help in future discussions with enforcing authorities.

The first step in any such sewer loss control program is to select sampling points. The decision as to how many sampling points should be used and where they should be located must be based on consideration of individual mill conditions.

The ideal system would provide sampling of the effluent from each individual mill operation. However, such an arrangement must be tempered both by economic consideration of the cost of the installation and by consideration of the existing sewer arrangement in the plant.

Probably the best plan is to start with a sampling station which measures and samples the entire plant discharge. If all the effluent does not presently discharge through an existing single sewer, the relative cost of additional master sampling stations should be compared with the cost of combining sewers.

In addition, the initial installation would provide for separate sampling stations for critical operations such as pulp washing, the beater room, and discharge from the savealls.

Records should then be kept of total sewer losses compared to losses accounted for by the separate stations. If the master station shows losses substantially in excess of the individual sampling stations, a careful survey should be made to determine the source of the additional unaccounted-for loss, and sampling stations installed on the individual sewers from the plant operation which has proved to be the source of the additional pollution.

In order to provide the information required to effectively control sewer losses, each sampling station must provide accurate information with regard to total flow by the station, must provide an accurate integration of total fiber and chemical losses that have passed the station, and must provide some sort of instantaneous alarm and record of sharp and sudden increases in the chemical concentration of the flowing material.

Fortunately, the tools for performing these functions are now readily available. The primary and most basic information which is required, the actual measurement of flow will, of course, be dependent upon the physical arrangements of each plant. Most sewers do not run full at all times, and therefore conventional differential type instruments using an orifice plate or Venturi tube are not usually applicable. Usually, however, it is possible to install a flume or weir in a sewer manhole and obtain substantially accurate measurements from one of these metering devices.

JOHN G. DOBSON, The Foxboro Co., Foxboro, Mass.

Figure 1 shows a typical installation of a V-notch weir in a rectangular sewer. The flow over such a weir is a function of the height over the weir, and therefore, by measuring the height and knowing the mathematical relationship, it is possible to calibrate a flowmeter directly in terms of million gallons per day, or other appropriate units, while the flowmeter is actually measuring the height in the stilling well. Such meters, reading directly in gallons per minute or million gallons per day, are readily available. If any substantial quantity of suspended matter is present in the material in the sewer, it is advisable to use a continuous purge of water into the stilling well to prevent settling of solids in the well.

The choice between a V-notch weir and a rectangular weir is dependent upon the available head, or, in other words, dependent upon the allowable loss across the measuring device and upon the range of expected flows. The various V-notch

measurement, by making an actual record of flows through a department's sewer. By equipping the flowmeter with an integrator, information is then available, both to management and to the operator, as to the total quantity of flow which has passed during a particular period, such as a shift, and also information as to when the peak flows occur.

Usually such flowmeters will be located at a point remote from the operating station of a particular department. For this reason it is advisable to provide a second record and indication of total flow, at a point within easy reach of the operator of the particular department. A flowmeter equipped with pneumatic transmission will provide such a record, at a point where the responsible operator can see it; whereas the same record, at some remote point, might be only a history of past mistakes instead of being a tool for the operator, to help him correct mistakes as they occur.

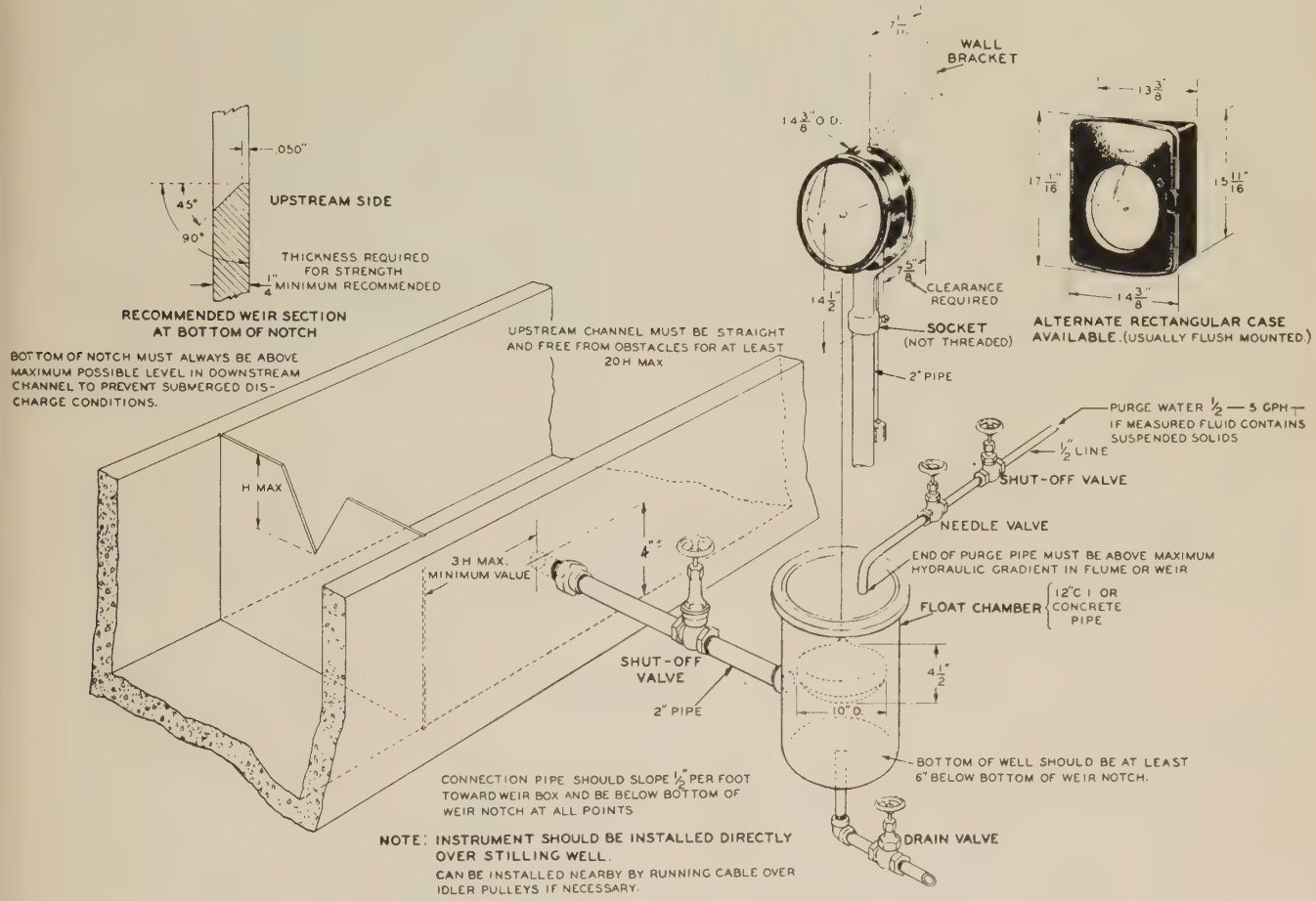
WALL BRACKET
14 3/8" O.D.
13 3/8"
17 1/16"
15 11/16"
14 3/8"
ALTERNATE RECTANGULAR CASE AVAILABLE. (USUALLY FLUSH MOUNTED.)
.050"
45°
90°
UPSTREAM SIDE
THICKNESS REQUIRED FOR STRENGTH
1/4" MINIMUM RECOMMENDED
RECOMMENDED WEIR SECTION AT BOTTOM OF NOTCH
BOTTOM OF NOTCH MUST ALWAYS BE ABOVE MAXIMUM POSSIBLE LEVEL IN DOWNSTREAM CHANNEL TO PREVENT SUBMERGED DISCHARGE CONDITIONS.
UPSTREAM CHANNEL MUST BE STRAIGHT AND FREE FROM OBSTACLES FOR AT LEAST 20H MAX
7 5/8" CLEARANCE REQUIRED
14 1/2"
SOCKET (NOT THREADED)
2" PIPE
PURGE WATER 1/2 — 5 GPH — IF MEASURED FLUID CONTAINS SUSPENDED SOLIDS
1/2" LINE
SHUT-OFF VALVE
NEEDLE VALVE
END OF PURGE PIPE MUST BE ABOVE MAXIMUM HYDRAULIC GRADIENT IN FLUME OR WEIR
FLOAT CHAMBER { 12"C.I. OR CONCRETE PIPE
H MAX
3H MAX. MINIMUM VALUE
4"
SHUT-OFF VALVE
2" PIPE
4 1/2"
10" O.
BOTTOM OF WELL SHOULD BE AT LEAST 6" BELOW BOTTOM OF WEIR NOTCH.
CONNECTION PIPE SHOULD SLOPE 1/2" PER FOOT TOWARD WEIR BOX AND BE BELOW BOTTOM OF WEIR NOTCH AT ALL POINTS
NOTE: INSTRUMENT SHOULD BE INSTALLED DIRECTLY OVER STILLING WELL.
CAN BE INSTALLED NEARBY BY RUNNING CABLE OVER IDLER PULLEYS IF NECESSARY.
DRAIN VALVE

Fig. 1. Recommended installation of flow metering equipment for weir measurements

weirs, as can be seen from Fig. 2 have a much wider rangeability. However, the rectangular weirs have a larger flow for a given loss and therefore are more suitable for installation in sewer lines where limited head loss is allowable.

If the head loss is still more limited, or substantial quantities of fiber or other suspended matter are in the waste leaving the mill, a Parshall flume as illustrated in Fig. 3 may be used to advantage. However, with proper operation of the mill there will not be sufficient suspended matter in the sewers to require the use of such a flume.

Various other primary devices for measurement of flow have been suggested and are available on the market. There are cases where these more expensive devices may be justified by special local conditions. However, in most cases the conventional weirs and flumes, which can be constructed locally at reasonable cost, will meet the requirements for

Even more important than accurate flow measurement to the over-all purpose of control of sewer losses is the obtaining of an accurate and representative sample of the waste being discharged. The taking of grab samples from a sewer at intervals is totally unsatisfactory, since these samples may be taken at times when representative conditions exist or may be taken at a moment when unrepresentative minimum or maximum conditions may exist.

Continuous samplers have been used. If the waste does not change particularly in quantity or quality, continuously operating sampling pumps may be satisfactory. However such steady conditions seldom exist, and it is almost always necessary to take a sample with the quantity corrected for flow conditions. There are two general methods of taking a sample which will be corrected for variation in flow rate. The two methods differ only in the frequency of obtaining the

sample, and the choice between the two is dictated by considerations of the minimum size of the sampling pump which can be used without running into clogging and valve difficulty, and of the maximum size of the sample which can reasonably be stored.

flow dropped to 500 g.p.m., the pump would operate 30 seconds out of every minute and if the flow dropped to 250 g.p.m., the pump would operate only 15 seconds out of every minute.

This method usually yields an excessively large sample unless an extremely small sampling pump can be used.

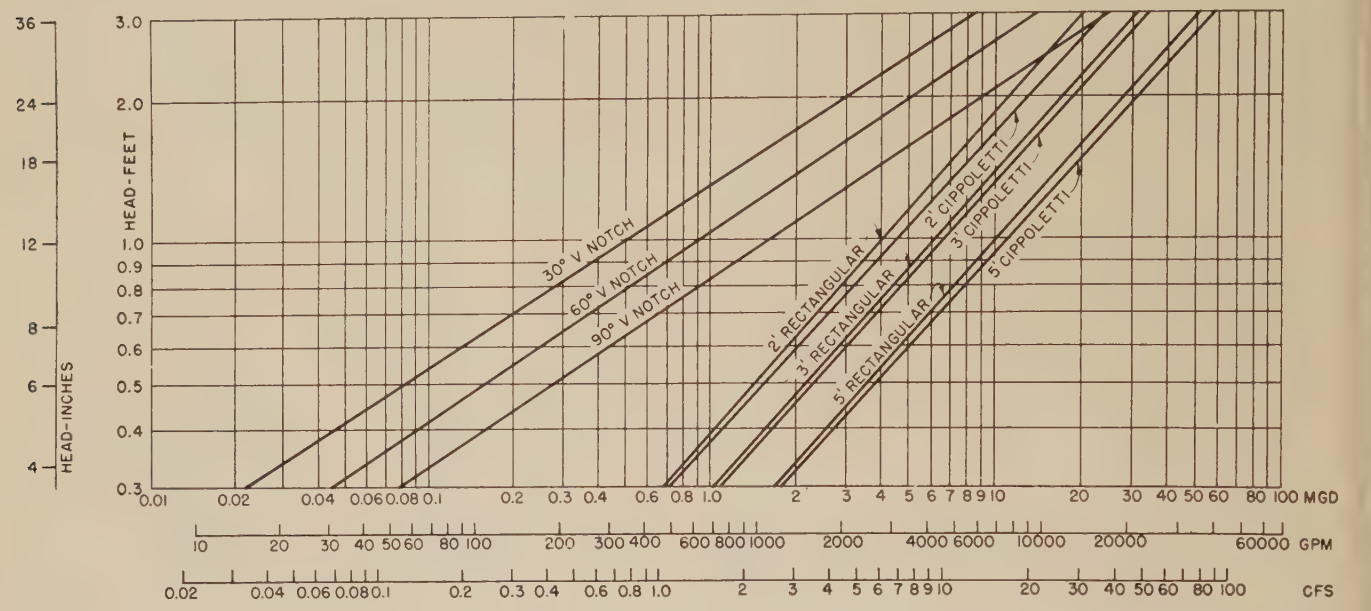

Fig. 2. Capacity curve for rectangular Cippoletti and V-notch weirs plotted against head across the weir

The first and preferable method is to run a portion of every 1-minute period. For instance if the sewer line has a maximum flow of 1000 g.p.m., the system would then be designed to operate the pump for 60 seconds out of each minute when the flow was 1000 gallons. However, if the

The second method is to operate the pump for a fixed period at the end of a given number of gallons of flow. Taking the same example, with the sewer running at a maximum capacity of 1000 g.p.m., the pump might be operated for 30 seconds every time a total flow of 500 g.p.m. had passed the measuring point. In this way the pump would operate for 30 seconds

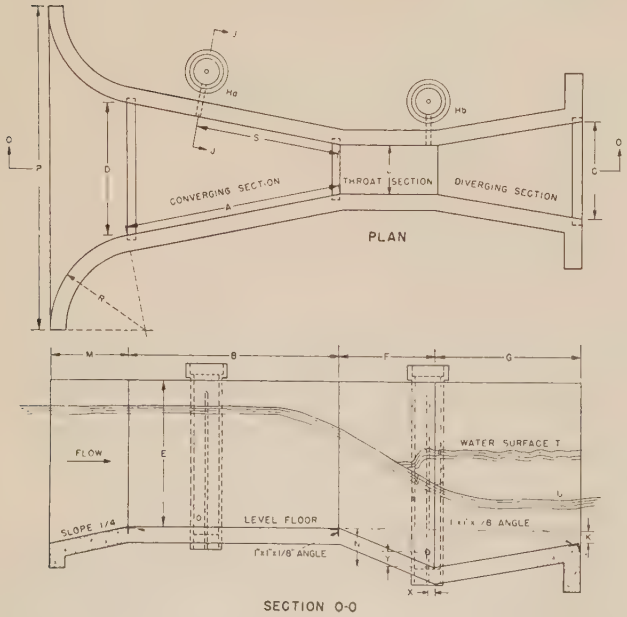

Fig. 3. Recommended design of Parshall flume for measurement of plant effluent

These may be installed in sewer manholes, sewer lines, or at pipe-line discharges.

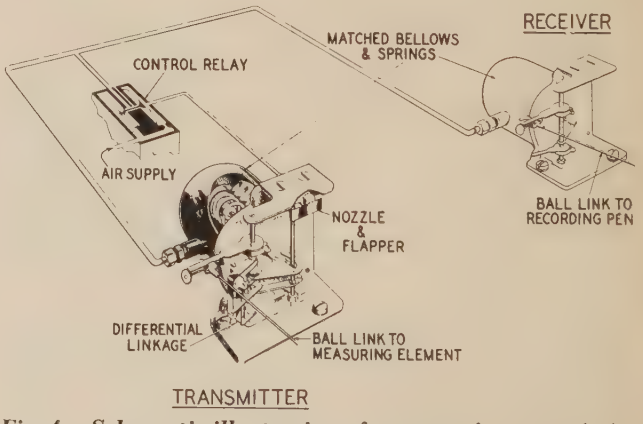

Fig. 4. Schematic illustration of pneumatic transmission system used to provide flow measurement record at location convenient for use by operator

out of 5 minutes when the sewer was running at maximum flow. When running at half-capacity, or 500 g.p.m., it would operate for 30 seconds out of 10 minutes; and when running at quarter-capacity, or 250 g.p.m., the pump would operate for 30 seconds out of every 20 minutes.

The limitations of this method are obvious since the sampling period is extended and flash conditions occurring

during the pump's idle periods may not be reflected in the integrated sample.

Whether this is important or not depends entirely upon the rate of change of the materials being discharged to the sewer.

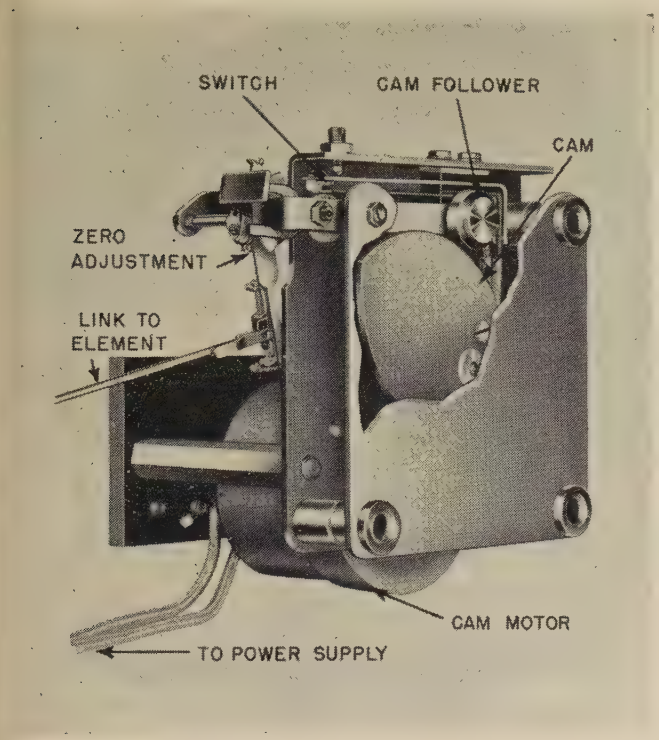

Fig. 5. Mechanism for providing period of pump operation proportional to flow to provide accurate sampling

The sample which is obtained in this manner is, of course, a truly integrated sample; and the total quantities of any particular constituent can be obtained by measuring the concentration of the integrated sample and multiplying it by the integrated flow which has occurred during the sampling period. Therefore, by routine chemical analysis, either by manual titration or by spectroscopic means, the concentration of the various constituents of interest to the mill may be determined and the losses evaluated. The usual kraft mill will be interested in the lime content, the soda content, and fiber content. If records are also being kept for later discussion with stream pollution authorities, it may be desirable to run B.O.D. by standard methods.

Ordinarily, in paper mill practice it is not necessary to take any particular precautions in the storage of the sample from the time it is pumped into the sampling container until it is taken to the laboratory. This is usually done at 8-hour periods, and therefore negligible amounts of change will occur in the sample. However, if the sample is stored over extended periods during hot weather, and B.O.D. is of interest, it is advisable to store the sample at below 40°F. in order to minimize bacteriological action during the storage period.

These samples, if properly stored, can provide another important kind of information to the operator of the paper mill. By careful examination of the fibers in the sample, it is possible to determine if any decomposed fiber is present in the waste coming from the mill, and in this way to determine whether dead areas with attendant stock degradation are occurring in the stock systems.

Actual equipment for operating the sampling pump is shown in Fig. 4. Essentially, the switch which is shown here is actuated for a portion of a 1-minute period depending upon the positioning of this arm. This arm is turned and positioned by the flowmeter, and therefore the "on" period of this switch is proportionate to the flow.

The first method of sampling discussed is done by merely placing a relay in series with these contacts and operating the pump from the relay contacts. In the second method of sampling these contacts actuate the motor of a small electric clock which is an integral part of a standard timer. The timer will make a contact when the flow totals a given number of gallons and, in turn, will actuate a second timer which will determine the running period of the pump.

The actual choice of the pump is a difficult problem.

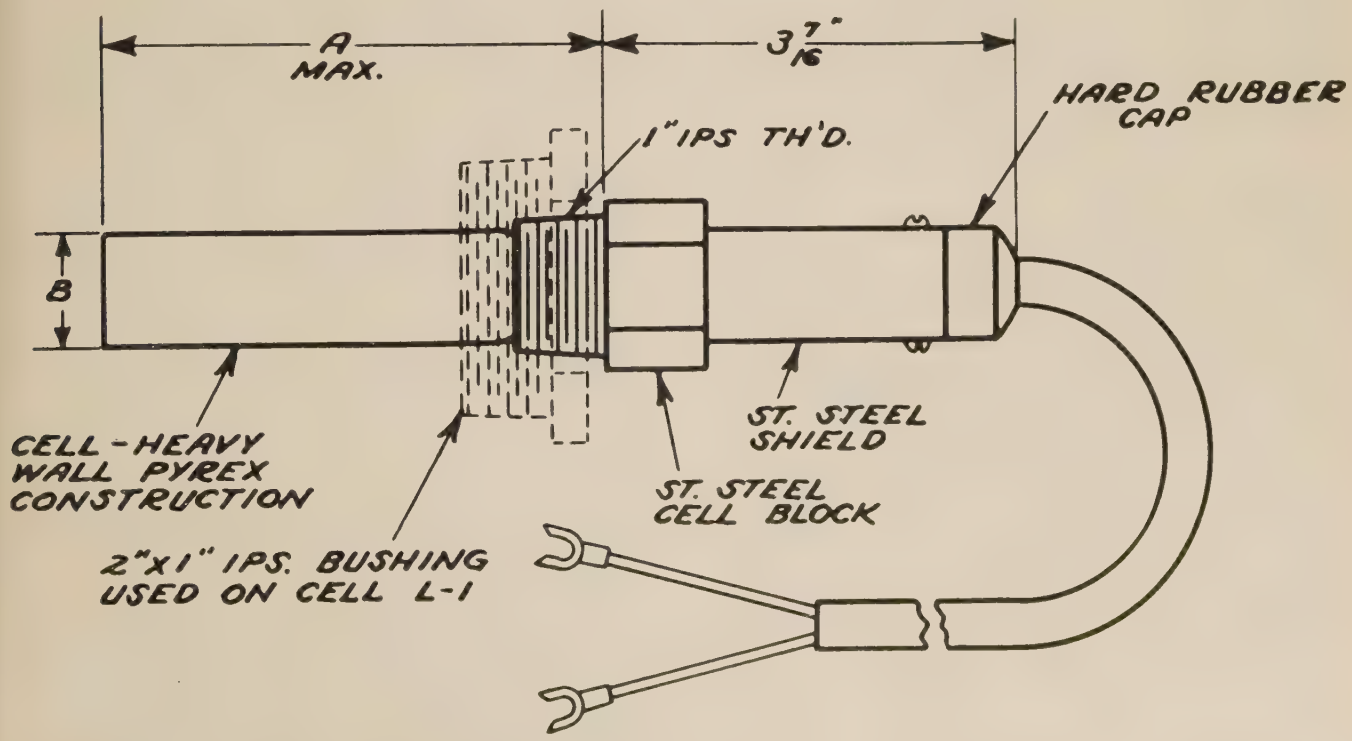

Fig. 6. Typical cell for determination of conductivity of plant effluent to determine changes in composition

There are various proportioning pumps, most of which have been designed for handling of reagent solutions, such as hypochlorite in water treatment, and which are satisfactory for this application if the waste is absolutely clear.

Pumps with ball-type seats of larger construction are satisfactory if small amounts of suspended solids are present. This is particularly true of the double valve pumps.

However, if substantial amounts of suspended solids are present in the waste, an Egyptian Wheel-type of pump seems to be the most satisfactory. These are available in limited design from several manufacturers or can be built locally.

The choice of a point for the suction of the pump is very important. It should be at a point where no stratification of the sewage is taking place, and should be so chosen that any suspended solids will be picked up in their proper proportions. One simple scheme is to place a reasonably constant level trough under the discharge from a weir and in this way obtain a constant level supply of material which has recently been mixed and agitated so that it is substantially uniform.

If the installation of pumps in this particular place is not reasonable it may be necessary to install agitation at some other point in order to produce a representative sample.

Composite samples obtained in this manner are, by their very nature, not available for immediate action when an operating irregularity occurs in the mill. Essentially a composite sample can only be taken at fairly infrequent intervals, say 8-hour periods, and can only be made available to the actual operator after the laboratory analysis has been completed.

As a result a shift operator will seldom know if an operating irregularity has occurred on a particular day until after he returns for the next day's operation.

Therefore, while such a sampling procedure is valuable for evaluating the total sewer losses and for watching trends in total sewer losses over a period, additional information must be made available to the operator if operating irregularities are to be corrected while they still exist.

Fortunately in the pulp mill a tool for this purpose is readily available. The electrical conductance of a solution is usually increased as the concentration of soluble electrolyte is increased. Therefore, in wash water going to a sewer, if the amount of salt cake or lime increases sharply the electrical conductance of that solution will similarly increase.

In the solution of a mixture of salts it is impossible to determine the actual concentration of one particular salt by the measurement of the electrical conductance because other salts, also varying in concentration, will mask the effect.

Therefore, conductivity instruments are not useful for actual determination of concentrations of lime and salt cake in the sewer.

However, any sudden increase in the conductivity of the solution is indicative of a sudden increase in one of the conductive constituents. A cell such as is shown in Fig. 5 can be immersed in the sewer line and connected to an instrument as illustrated. This instrument measures the conductivity at 1000 cycles and therefore eliminates polarization errors in the conductivity measurement. The instrument itself will record the conductivity and indicate any sharp changes, and can be set up so as to give an electric alarm if the conductivity increases beyond a certain predetermined value.

Any mill can determine within a short period of experience what the normal operating conductivity of its sewage is and provide for an alarm if this is exceeded. In this way an operator is immediately warned when the losses exceed what is considered reasonable.

In some wastes, particularly in the drainage ditches in the acid plant of a sulphite mill, the measurement of pH may be more sensitive to small leaks in valve and pump packing glands than would conductivity. pH is also a readily available measurement and can be set up on the same basis as conductivity.

Sewer losses are real dollars going down the drain. By proper surveying and proper policing they can be controlled. Their control not only saves dollars in chemicals which otherwise are wasted, but also reduces the total pollutional load which is being discharged to the stream (or to a treatment plant if such proves necessary). The program, however, to be successful, must be complete. It must have accurate flow data from various departments, and this flow data must be in the hands of the operator who can make use of it. It must provide accurate samples, properly integrated in terms of flow, which samples should be carefully analyzed in the laboratory and records maintained so that long-period trends can be established.

In addition, instruments must be installed which will give immediate alarms to the operators when operating irregularities are occurring. This information must reach them in time for them to make corrections before substantial amounts of chemicals are lost.

The program is expensive, but there are real dollars to be saved by it, and the dividend of reducing pollution is thrown in as a profit for which no additional expenditure need be made.

Presented at the Annual Meeting of the Technical Association of the Pulp and Paper Industry, New York, N. Y., Feb. 20–23, 1950.

Adhesion of Sodium Silicate to Metal

JOHN H. WILLS and ROBERT H. SAMS

SELDOM IN the history of the development of adhesives is a decrease in the strength of adhesion reported with satisfaction. It is also seldom that an adhesive is called upon to stick to one surface and not to another. This development of selective adhesion is manifestly difficult and the man who tries it is truly on the horns of a dilemma.

Sodium silicate bonds are not only stiff and strong but they also attach themselves strongly to metal as well as to paper. The latter is highly desirable but it has long been realized that the box maker would benefit if the strength of the silicate bond to the metal hot plates could be reduced. With this in mind the problem has been studied by various workers for at least fifteen years. One technique has been to add various materials to the silicate with the hope that one might weaken the attachment to metal without reducing the usual high strength of the silicate-paper bond. To our knowledge, more than two hundred such additives have been tried with little success. Even more numerous tests of protective coatings directly on the metal have failed to provide the complete answer.

Recently it has been found that additions of a rosin-maleic anhydride condensation product sold by Monsanto Chemical Company under the trade name of Mersize gives results greatly superior to anything previously investigated and sufficiently good to be of real interest to the boxmaker. This was demonstrated in an experiment where drops of sodium silicate adhesives were placed on metal surfaces which had been heated to 330°F. The results were rated according to visual and manual comparison on a basis of 1 to 10, where the straight silicate adhesive which was hardest to remove was given a rating of 10 and a mixture which separated cleanly from the metal with only a gentle tap of a scraper tool was rated as 1. Additions to silicate of inorganic materials such as sodium chloride, ammonium phosphate, hydrogen peroxide, and many others showed no reduction in

JOHN H. WILLS AND ROBERT H. SAMS, Philadelphia Quartz Co., Philadelphia Pa.

bond strength to the metal. Easily decomposed materials such as urea, para-formaldehyde, and Porophor N (a complex compound used by the Germans in formulating their rubber compositions) gave no improvement. Several natural products such as spruce extract, pine tar oil, pine oil, and soy bean oil were of little help. Spruce extract showed a decrease to as low as a value of 6. Additions of alkyl sodium sulphonate, sulphonated castor oil, or triethanolamine were sufficiently helpful to receive a rating of 4. A terpene maleic anhydride and a linoleic acid-maleic anhydride, both related to Mersize gave silicate combinations which could be rated as number two. Mersize with silicate definitely qualified for the number 1 position.

This experience has been confirmed by field tests where plant operators have reported that the use of Mersize with silicate reduces the clean-up effort to a very brief pass over the hot-plate section with a scraper. The slight adhesive residue remaining on the metal is thereafter worn away by the abrasion from the paper web passing over it.

The reason for the superiority of Mersize over other additives is a matter of conjecture. It is well known that sodium silicates on hot metal will puff to form a layer honey-combed with air bubbles. Such films are used as insulation directly, or as paints which will puff when exposed to fire and thus protect the under surface. Examination of a cross section of a Mersize-silicate film on metal, using a projection of a Faxfilm impression, showed that the bubbles at the interface were much smaller than when regular silicate adhesives were used. Presumably the cell wall area is greater and the walls themselves must be thinner. While the exact mechanism of the action is not known it has been established that Mersize does not decompose at the temperatures normally used for hot plates (330°F.), nor is there excessive boiling. Another possible factor is in the nature of the Mersize molecule. It is known that it has one portion with a high dipole moment which would cause orientation at the metal surface, and it also has a high concentration of acid groups to react with the metal. Thus it is probable that a protective film of Mersize is formed on the surface of the metal.

The ease with which the paper web wears away the residue left on the hot plate after tapping the main deposit with a scraper is of critical importance to the mill operator. This was investigated by measuring the force necessary to crush the honey-combed structure just above the interface between the metal and the dried film. Results were obtained in triplicate and give only a rough idea of the effect but as recorded in the following table they do show that 0.1% Mersize reduces the resistance by at least 66%. The effect is not greatly influenced by the concentration or ratio of the silicate nor does the concentration of Mersize above about 0.1% appear to change the force required. However, experience shows that the film is much more easily tapped free from the metal surface if the amount used is in the neighborhood of 0.5–0.75%.

Table I. Resistance to Shear of "N" Silicate Adhesive Containing 0.1% of Compounds Related to Mersize

	Pounds
No additive	30
Mersize, regular	8
Mersize, 100% neutralized	9
Sulphonated rosin, sodium salt	10
Sulphonated rosin	12
Sulphonated Mersize	11
Terpene-maleic anhydride	12
Linoleic acid-maleic anhydride	11
Styrene-maleic anhydride	13

The addition of Mersize to silicate is best accomplished by pouring the Mersize into part of the silicate solution and then adding the remaining silicate. In that way the tacky Mersize is prevented from touching the walls of the container and the final addition of the silicate of soda helps to disperse the viscous additive. Dispersion will eventually occur merely by diffusion but it is aided by agitating the mixture with a paddle or with a compressed air lance or by warming. Even these precautions are scarcely necessary with the improved Mersize now available and known as Mersize CD-2. With the earlier form it was helpful to add small amounts of caustic soda to a slightly diluted commercial Mersize preparation before introducing it into the silicate.

The perfect answer to this difficult problem is still to be found but the current experience with Mersize is a long step ahead. Although the work is not as precise nor as detailed as desired, it does show that the bond between sodium silicate and hot metals can be reduced to a low value. The dried film remaining on the metal can now be weakened sufficiently to wear away if the upper hard, unpuffed layers are first removed and it requires only a light touch with a scraping tool to accomplish this. Experiments have shown that the bond to paper is as strong as ever.

LETTERS TO THE EDITOR

Drying of Paper

To the Editor, *Tappi*

Tappi 33, no. 5, 64–68 May, 1950, printed the talk by A. E. Montgomery entitled "The Drying of Paper." I would like to add a few words to the discussion which followed:

During the discussion Mr. Montgomery asked: "Why are desuperheaters used?" (referring to the steam entering the driers). The answers to this question were not very specific, so I would like to suggest two items which might be considered:

1. The use of saturated steam gives rise to a constant temperature on the steam side. The steam gives up heat by condensation and then two phases are present giving one degree of freedom of the system. Since the temperature thus is determined by the existing pressure this provides a means of temperature control.
2. Heat is transferred from condensing steam giving rise to either film type or dropwise condensation depending on the purity of the steam.

If superheated steam is used on the steam side the following conditions would obtain:

1. The temperature on the steam side would not be constant since the superheated steam would give up sensible heat before condensation. This would cause an irregular flow of heat.
2. At the points where the superheated steam gives up its sensible heat the heat transfer coefficients would be small compared with those existing at points where condensation occurs. This would create an uneven rate of heat flow.

It might well be argued that these two factors tend to cancel each other; yet it is expected that the heat-transfer coefficient effect would predominate. Based on these remarks I would like to answer Mr. Montgemery that the use of desuperheaters would enhance an even rate of heat flow, all other conditions remaining constant.

As an embryonic pulp and paper engineer I am in no position to evaluate the practical utility of these factors, yet, I wish to bring them to your attention for consideration. Let me close the letter expressing my appreciation to Mr. Montgomery for his talk.

P. Bro, *Dept. of Chemical Engineering*
Yale University, New Haven, Conn.

Response by Mr. Montgomery

I have read with interest the letter written you by P. Bro on June 3, copy of which you sent me. His comments are well taken and I agree with everything he says.

This morning I looked up the report of the discussion as it appeared in *Tappi* on page 66A of the May issue, and as I read this I can see plenty of room for question. Of course the discussion was long and the report of the discussion is tremendously condensed. For example, in one place I am reported to have said, "Dr. McAdams in his text on 'Heat Transfer' says they give slight or no advantage. They do make control more difficult." What I actually said or intended to say was that Dr. McAdams says that superheat gives slight or no advantage and I then commented that superheat makes control more difficult.

I was trying to find out whether anyone at the meeting had actually had experience with superheated steam which later had been desuperheated and could tell us the results. No one did have such exact information. Mr. Bro may be interested in knowing that actual claims are entirely conflicting. I know of some people who have had distinct improvement, or thought they had, when they desuperheated, whereas others say they could detect no difference.

A. E. Montgomery
J. O. Ross Engineering Co.

A Compliment

To the Editor, *Tappi*

I have just had the occasion to look over *Tappi*, May, 1950, Volume 33, No. 5, and to express an opinion to you that this is one of the best technical publications that I have had occasion to read for some time.

I want to congratulate you on the manner in which *Tappi* is being handled. It is a great pleasure to see that the Technical Publication of an industry so important as the pulp and paper industry is coming out in such good shape and is in such good hands. My congratulations.

Wm. Carter, *Vice-President*
Canadian Ingersoll-Rand Co.

Routine Control Methods

To the Editor, *Tappi*

We wish to express our appreciation to the Association and its committee for the Routine Mill Control Methods mailing just received.

Test RC-67 indicates the advantages of using lactic acid solutions to speed the results of flotation sizing tests on hard-sized papers and boards. For several years we have used 8.5% lactic acid test solution on a sheet having a minimum test specification of 60 minutes water flotation. However, we have found the use of antifoaming agents in the machine system not only reduce foaming, but also act as wetting agents, reducing the water flotation test and drastically altering the correlation between the lactic acid and plain water tests. Results on many types of antifoaming agents have shown the reduction in sizing and altered correlation to be characteristic of materials effective in controlling foam and apparently especially those materials having active groups such as acid or hydroxyl groups present or potential. Nonactive types such as petroleum hydrocarbons have little effect on sizing or correlation and, unfortunately, little effect on foam. If the concentration of the defoamer can be held constant in the machine system, a valid correlation can be established at least to the extent of being sufficiently indicative of changes in degree of sizing to materially aid in preventing manufacture of off specification material.

We offer the above observations with the idea of perhaps assisting someone else in recognizing the effects of antifoaming agents on the sizing itself and on correlation of the tests and thus arriving at satisfactory control more easily than we did.

F. H. Bopps, *Asst. Plant Manager*
American Can Co., Constantine, Mich.

To the Editor, *Tappi*

The new Routine Control Methods received are a useful innovation. Methods identical or similar to RC-1, 8, 11, 14, 32, 33, 42, 43, 50, 53, and 74 have been used by us for a considerable time. Referring to RC-53, we also use ammonium thiocyanate solution in the presence of hydrochloric acid for detecting iron in paper. Regarding RC-11, particularly where only very small samples are available, we make use of the old biuret test for the qualitative detection of proteins. The sample is soaked for a few minutes in 2–5% copper sulphate solution, dried and then spotted, with 1% caustic soda. The characteristic reddish-purple color which develops in the presence of proteins appears to be a reliable indication of their presence.

R. F. C. Holness
Waterlow & Sons Ltd.
London, England

Ed. Note: The two preceding letters are appreciated by TAPPI Routine Control Methods Committee. The Association is anxious to expand this service and can do so only if members will contribute methods that have been found to be useful and reliable. A review of the recent mailing will indicate, in general, the type of methods that are desired. Comments are requested also on the methods that have been published. Comments and methods should be sent to the Secretary, Technical Association of the Pulp and Paper Industry, 122 East 42nd St., New York, N. Y.

EMPLOYMENT SERVICE

Positions Open

P74-50. Paper engineer. Outstanding man for research and development on nonwoven fabrics. Southern location. Permanent position with excellent future. In replying give full background of training and experience and salary expected. Our employees are informed of this advertisement.

P75-50. Chemical or engineering graduate, 28–35 years old, with mill or technical board or paper mill experience, for sales service and marketing of well-established sizing products. Applicant's headquarters preferably in Chicago area, for traveling in Middle States, surrounding Lake Michigan. Excellent opportunity with nationally known company having country-wide manufacturing and office points. Write full qualifications and references including recent snapshot.

P76-50. Laminating and coating foreman. Experience with knowledge of hot melt, solvent and glue laminations and coating, to take complete charge of a plant in Holyoke, Mass. Give experience details.

P77-50. Chemist. Prefer man with five or more years' industrial laboratory and practical experience. Must have knowledge of resins, resin analysis, functional properties and their application to paper products. Salary commensurate with capabilities.

P78-50. Paper research chemist. Established organization desires research man for laboratory engaged in development of specialty products. Ph.D. desirable but not essential. Give details of education, experience and salary requirements. Location New York City.

Positions Wanted

E76-50. Engineer, 29, family. B.S. degree. Four years' service and sales engineer for instrumentation in pulp and paper mills. Also production control and marine power plant experience. Desires position in either production or mechanical department of northern pulp and paper mill.

E77-50. Graduate chemist, 34, married. Seeks position with northern paper or textile manufacturer. Interested in paper twisting and conversion of paper for textile uses; 12 years' experience in textile and allied industry.

E78-50. Present section leader in pulp research desires administrative or advisory technical position. 21 years' fruitful development research in acid and alkaline pulping and bleaching. Particular qualifications in solution of special mill problems. Not interested in South or metropolitan locations.

Thermal Conductivity of Structural Insulating Board*

TAPPI Tentative Standard T 1000 m-50

THIS METHOD IS the standard guarded hot plate method of the American Society for Testing Materials for determining thermal conductivity (*1*). It has been arranged to conform to TAPPI format and somewhat condensed for application to flat slabs of homogeneous structural insulating board as defined by all classifications of Commerical Standards CS-42 and CS-112 (*2*), A.S.T.M. Designation C-208-48 (*3*), and Federal Specification LLL-F-321b (*4*).

SCOPE AND LIMITATIONS

For practical purposes, the method is limited to materials having conductivities not in excess of 5.0 B.t.u. per inch per square foot per hour per degree F. It is further limited to thermal conductivity tests between the extreme temperatures of −50°F. and upward of over 212°F., or between mean temperatures above approximately 0°F.

Because of the requirements prescribed as to conditions under which tests shall be made, the conductivity coefficients obtained will not necessarily be the values pertaining under all service conditions. For example, the coefficients are obtained on dry specimens, whereas in service such a condition will seldom be realized.

The method is intended for homogeneous materials in the form of flat slabs. For satisfactory results, the principles governing the size, construction, and use of apparatus for the test described in this method should be followed. If the results are to be reported as having been obtained by this method, then all the requirements prescribed in this method shall be met.

It is impossible in a method of this type to establish details of construction and procedure covering all contingencies so that it can be followed by a nontechnical person; consequently, technical knowledge on the part of those using the method concerning the theory of heat flow, temperature measurement, and general testing practices is necessary.

SYMBOLS AND DEFINITIONS

The symbols used herein have the following significance:

Q = total quantity of heat transferred
T = time
$q = Q/T$ = rate of heat flow
A = actual area normal to the path of heat flow (flat surface)
k = thermal conductivity
t_1 = temperature of the hot surface
t_2 = temperature of the cold surface
L = length of path of heat flow (thickness)
R = thermal resistance (degrees, per unit of heat transferred per unit of time)
$C = 1/R$ = thermal conductance (heat transferred per unit time, per degree)

* This method has been approved as a tentative standard by the Structural Fibrous Materials Committee. Criticisms are earnestly requested and should be sent to R. G. Macdonald, Secretary, Technical Association of the Pulp and Paper Industry, 122 E. 42nd St., New York 17, N. Y.

Thermal Conductivity

The thermal conductivity of a homogeneous material is the rate of heat flow, under steady conditions, through unit area, per unit temperature gradient in the direction perpendicular to the area. It is calculated as follows:

$$k = \frac{QL}{AT(t_1 - t_2)} = \frac{qL}{A(t_1 - t_2)} \qquad (1)$$

Note: Thermal conductivity is usually and for structural insulating board should always be expressed in English units as B.t.u. per square foot per hour per degree F. for a thickness of 1 inch. This may be expressed mathematically as B.t.u. in. per hr. sq. ft. deg. F., or B.t.u. hr.$^{-1}$ ft.$^{-2}$ deg. F.$^{-1}$ in., or in various other forms. Other units are also found in the literature, and conversion factors are given in Table I.

Thermal Conductance

The thermal conductance of a body between two specified isothermal surfaces is equal to the rate of heat flow through the body, per unit difference between the temperatures of the isothermal surfaces under consideration, and is calculated as follows:

$$C = \frac{1}{R} = \frac{q}{(t_1 - t_2)} \qquad (2)$$

Note: Thermal conductance is usually and for structural insulating board should always be expressed in English units as B.t.u. per hr. per deg. F. temperature difference between the two surfaces. The term C has largely been eliminated in favor of the expression $1/R$. The heating and ventilating engineer, however, dealing with compound walls, makes considerable use of the term unit conductance or $C/$ unit area, and therefore continued the use of the term C for conductance. (This must not be confused with the abbreviation C. for Centigrade.)

CONDUCTIVITY-MEAN TEMPERATURE RELATIONSHIP

The thermal conductivity-mean temperature relationship shall be obtained from curves resulting from plotting the thermal conductivity vs. mean temperature for each pair of specimens. The average thermal conductivity at any desired mean temperature may be obtained from these curves by averaging the individual values from each curve at that temperature. By obtaining several such average thermal conductivities at different mean temperatures, an average thermal conductivity-mean temperature curve may be drawn for the sample.

APPARATUS

(*a*) It is not intended in this method to include detailed requirements for construction or operation of any particular guarded hot plate for determining conductivity values (see note). Any plate conforming to the limitations prescribed in the following paragraphs (*b*) to (*k*), inclusive, will be satisfactory.

Note: For convenience of new workers in the field, A.S.T.M. has arranged to make available complete directions and specifi-

Table I. Conversion Factors for Thermal Conductivity

	Cal. cm. per sec. per sq. cm. per deg. C.	*Watts cm. per sq. cm. per deg. C.*	*Kg.-cal. m. per hr. per sq. m. per deg. C.*	*B.t.u. in. per sq. ft. per hr. per deg. F.*
1 cal. cm. per sec. per sq. cm. per deg. C.	1	4.185	360	2903
1 watt cm. per sq. cm. per deg. C.	0.2389	1	86.00	693.7
1 kg.-cal. m. per hr. per sq. m. per deg. C.	0.002778	0.01163	1	8.064
1 B.t.u. in. per sq. ft. per hr. per deg. F.	0.0003445	0.001442	0.1240	1

cations for building and use of three typical guarded hot plates complying with the requirements of this method and known as the National Bureau of Standards plate, the National Research Council plate, and the Alundum plate. (Detailed information regarding each of these is available from the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.) The first two plates are square and have metal surface plates. The Alundum plate is built with heating coils molded into alundum cement so as to give a circular plate with alundum cement faces. It is advantageous for use in the moderate high-temperature field.

(*b*) The general features of the guarded hot plate are shown in Fig. 1. The plates are usually square, but round ones are occasionally used. The term guarded hot plate is applied to the entire assembled apparatus, including heating unit, cooling units, and edge insulation. The heating unit consists of a central heater and central surface plates. The guard section consists of one or more guard heaters and the guard surface plates. The surface plates are usually made of noncorrosive, highly conducting metal; however, the central section and the guard section of the heating unit may each be molded of alundum cement or other materials of low conductivity, so as to include both the heater and the surface plates (see note).

Note: The surface of the heater plates and cooling plates should be finished to as nearly a true plane as possible and should be checked periodically. Variations over 90% of the surface should be nil, with maximum variations on any portion of the surface not exceeding 0.005 inch.

(*c*) In the design of the guarded hot plate for testing materials for use in any particular temperature or conductivity range, due consideration shall be given to the materials used in the construction of the hot plate with respect to their performance at the temperature to which the plate will be subjected. Consideration shall also be given to the rate at which heat must be supplied and absorbed by the heater and the coolers in designing the electrical resistance and current-carrying capacity of the heating element. Otherwise, no particular variations need be made in the design of hot plates for use in determining the conductivity of materials between such widely differing mean temperatures as 0 and 1200° F.

(*d*) Heating units having metallic surfaces shall have a definite separation or air gap not greater than $^1/_8$ inch between the measuring area of the central surface plate and the guard surface plates. These plates shall have highly emissive surfaces. The separation between the main central section and the guard section of the heating units shall not exceed $^3/_4$ inch, and this separation is allowable only if the spacing bars on either side of the plate separation are of heavy copper in order to distribute the heat on the surface plates. In all other cases the separation shall not exceed $^1/_8$ inch. The test area shall be calculated from the center of one separation to the center of the other separation across the central surface of the plate.

Note: In putting a new heating unit into operation for the first time, take care to insure that the two faces of the heating unit maintain essentially equal temperature throughout the temperature range of the apparatus.

(*e*) The heater of the guarded hot plate shall be pro-

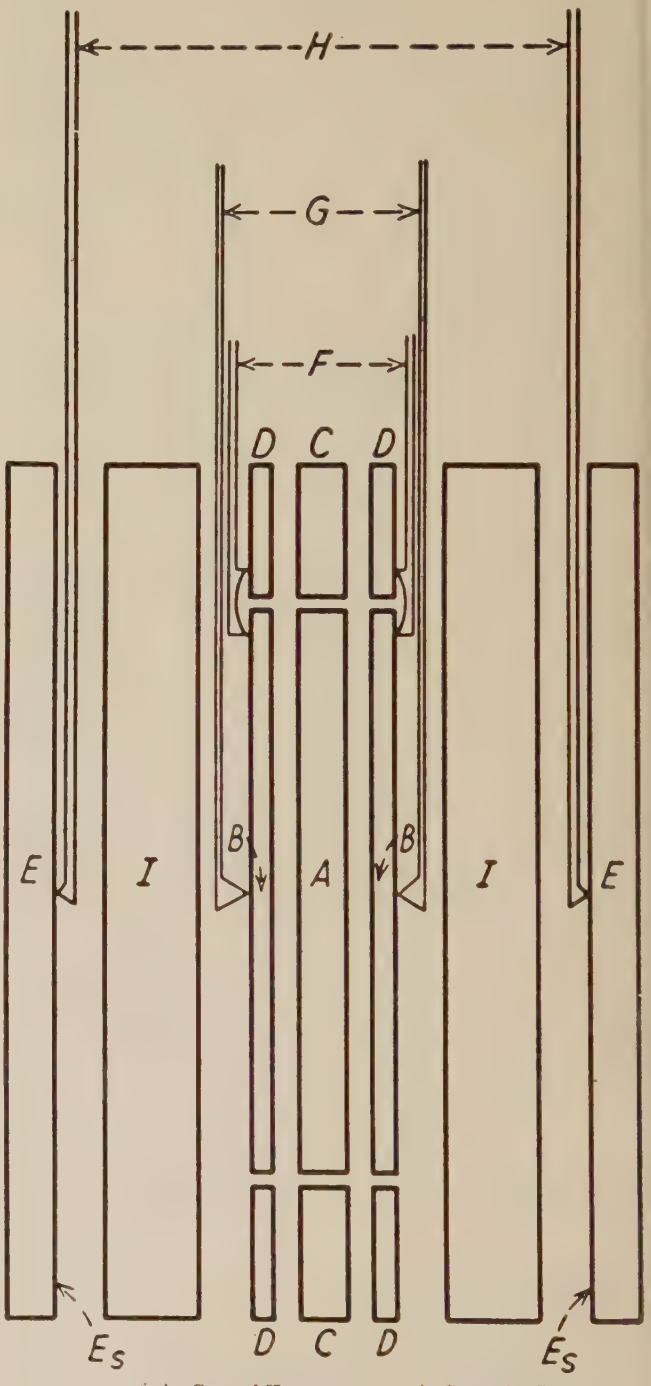

Fig. 1.—General features of the guarded hot plate.

vided with at least two thermocouples on each face of the central surface plate, and at least two thermocouples on each face of the guard surface plate, located at opposite edges. These may be read either individually to indicate any temperature difference that may exist between the central and guard surface plates or they may be connected differentially, and thus indicate such temperature difference directly. The differential method of connecting the center-to-guard thermocouples is preferable as it is more sensitive. When the thermocouples are to be read individually, they may be peened, welded, or soldered to the surface plates. When they are to be connected differentially, they must be electrically insulated from each other. This may be done by installing the thermocouples in shallow grooves in the surface plates with an electrically insulating cement, so that the bead of each thermocouple is in the plane of the plate's surface.

(*f*) The cooling units shall have the same surface dimensions as the heating unit. They may consist of metallic plates cooled by a fluid, electrically heated plates maintained at temperatures below that of the heating unit or thermal insulation applied to the cool surface of the test specimen, depending upon the mean temperature desired.

(*g*) The edge insulation may be of any convenient loose-fill or blanket-type insulating material to reduce edge losses from the heater plate, test specimens, and cooling plates. It shall be of such thickness that the resistance to edge losses shall be at least twice and preferably three or more times the thermal resistance of the specimen in the direction of normal heat flow.

(*h*) For structural insulating board, the surface temperatures of the test specimen shall be determined by means of thermocouples mounted in the hot and cold plates. Both the hot and cold plates shall each be supplied with at least two thermocouples mounted in the surfaces of the plates as described above in paragraph (*e*). Preferably, the thermocouples should be insulated from the plates and read differentially between the hot and cold plates, the surface temperatures of the specimen being taken as those of the plate surfaces in contact with it.

(*i*) The thermocouples mounted in the surfaces of the plates shall be made of wire not larger than No. 23 A.w.g.

(*j*) A potentiometer having a sensitivity of 5 microvolts or less shall be used for all measurements of electromotive force.

(*k*) Means shall be provided for measuring the distance between surfaces of the hot and cold plates when the specimens are in place.

TEST SPECIMEN

The sample shall be selected so as to provide two specimens as nearly identical as possible and of such size as to completely cover the heating unit. They shall be of sufficient thickness to give a true average representation of the material. Since excessive thickness of the specimens and consequent excessive dimensions of the area of the guarded hot plate unnecessarily complicate the manipulation of the test, the specimens should not be too thick. Their thickness shall be great enough to allow a sufficiently accurate measurement of this dimension to calculate the thermal conductivity.

The relationship between the maximum thickness of the test specimen and the minimum dimensions of the guarded hot plate shall be as follows:

Maximum thickness of test specimen, inches	*Minimum linear surface dimensions of guarded hot plate (square or round) inches*	
	Central section of test area of heating unit	*Guard section or guard area of heating unit*
1	4	1 1/2
1 1/2	8	2 1/4
2	12	3
(4	12	6)

The samples from which the test specimens are to be taken shall be weighed and measured for thickness and other dimensions in the as-received condition and immediately after drying to constant weight at 215 ± 2°F. (If this degree of heat is objectionable, they may be dried in a desiccator at 120 to 140°F.). Record the as-received and the dry weights of the sample and the necessary physical measurements for calculation of the density before and after drying.

The linear dimensions of each sample shall be measured to the closest 1/64 inch on each surface. Each measurement shall be not greater than 4 inches apart. The thickness shall be measured by means of a noncompressing micrometer or thickness gage having contacting surfaces not greater than 5/8 inch in diameter. Measurements shall be made not more than 4 inches apart and not less than 1 inch from the outer edges around the complete periphery of each sample.

Each sample, dried and conditioned as described above shall be cut into two specimens of correct size depending on the average thickness of the sample and linear dimensions of central and guard sections of the heating units of the guarded hot plate in use. The specimens shall be cut without compression, molding, or other distortion of thickness.

The thickness of the specimens as tested shall be as close as possible to the thickness of the original dry samples from which obtained. Specimens of greater thickness than allowable shall be reduced to the maximum allowable thickness by minimum sandpapering or other suitable means.

The surfaces of each test specimen shall be as plane as possible for intimate contact between specimens and plates. Specimens having individual thickness measurements greater than ±0.010 inch of the average specimen thickness, due to irregular or nonparallel surfaces, shall be corrected to within ±0.010 inch tolerance by minimum sandpapering or other suitable means. The average thickness of each specimen cut from a single sample and to be used as a pair in testing of that sample shall not differ by more than 0.010 inch.

Pairs of specimens from each sample, prepared in accordance with the above procedure, shall be redried to constant weight and immediately placed in a desiccator at room temperature in order to avoid moisture regain prior to testing.

PROCEDURE

The three or four tests described shall be made on each of three pairs of specimens representing three separate samples. As each pair is removed from the desiccator for testing, each specimen shall be carefully reweighed to the nearest 0.01 gram and remeasured for

thickness as described above, then placed immediately in the guarded hot plate. The assembly of the plates shall be carefully controlled so that the distance between surfaces of the hot and cold plates is exactly equal to the average thickness of the test specimens. This is a necessary precaution for structural insulating board in order to avoid errors due to improper contact of the surfaces and to compression or reduction in thickness.

Make at least three and preferably four determinations on each specimen at mean temperatures which will cover the range over which the material is to be used. For any test, the temperature difference across the specimen shall be at least 40°F. The above mean temperatures of the specimens shall differ from each other by at least 30°F. One test shall be at a mean temperature not higher than 60°F. and one not less than 170°F. The atmosphere surrounding the test equipment shall have a dew-point temperature not higher than the coolest part of any surface or material in, or forming a part of, the test apparatus.

Since thermocouples mounted in the surfaces are used to determine surface temperature, the thickness of the test specimen is taken as the distance between the surfaces of the hot and cold plates. When the specimen is in place in the apparatus, the plates shall be adjusted with clamps or pressure so that this thickness shall be within ±0.010 inch of the average specimen thickness prior to testing. Upon completion of the test, the specimens shall be reweighed immediately and the weight recorded. After each test, the specimens shall be redried to constant weight and cooled to room temperature in a desiccator prior to retesting at the next mean temperature.

Supply the heating element of the central heater with electrical energy regulated to give the desired temperature gradient through the test specimen and held constant within ±0.25%, means being provided to measure this energy. Automatic regulation is recommended; where this is not available, regulate the energy input by manual adjustment. Then adjust the rate of input to the guard ring so that the temperature difference between the center and guard surface plates during the 5-hour test observation period shall be not greater than ±0.1% of the average temperature drop between the two halves of the specimen, as determined by the differential or surface thermocouples.

Adjust the cooling units so that the temperature drops through the two test specimens do not differ by more than 1.0%.

After steady state has been reached, continue the test, making the necessary observations to determine temperature difference, center-to-guard thermal balance, and heat input, until successive observations made at intervals of not more than 1 hour, over a period of 5 hours, give thermal conductivity values that are constant to within 1.0%. Upon completion of the test, reweigh the specimen and record the weight.

CALCULATIONS

Calculate the density of the dried sample, the original moisture content and the moisture regain during the test as follows:

Let

D = density after drying (pounds per cubic foot or grams per cubic centimeter)
M = moisture in sample as-received (pounds per cubic foot or grams per cubic centimeter)
I = moisture regain (pound per cubic foot or grams per cubic centimeter)
E = weight of sample after drying (pounds or grams)
B = volume of sample after drying (cubic feet or cubic centimeters)
H = weight of sample as received (pounds or grams)
F = weight of specimen prior to test (pounds or grams)
G = weight of specimen after test (pounds or grams)

then

$$D = \frac{E}{B} \tag{3}$$

$$M = \frac{H - E}{B} \tag{4}$$

and

$$I = (G - F) \times \frac{E}{F} \times \frac{1}{B} = \frac{E(G - F)}{BF} \tag{5}$$

Calculate the thermal conductivity according to equation (1) previously given under Thermal Conductivity.

REPORT

The complete report of the results of each test shall include:

1. Name and any other identification of the material.
2. Average thickness of each pair of specimens tested.
3. Dry weight of sample before test.
4. Density of dried sample (before test).
5. Moisture in sample as-received.
6. Moisture regain during test of each pair of specimens.
7. Mean temperature range.
8. Maximum hot-surface temperature.
9. Minimum cold-surface temperature.
10. Mean temperature of each test.
11. Heat input in B.t.u. per sq. ft. per hour of each test.
12. Thermal conductivity of each test.

Note: If desired, the moisture in the sample as-received (item 5) and the moisture regain (item 6) may be reported also as percentages of the weight of the *dried* sample. These are calculated as follows:

Let

PM = % moisture in sample as-received (dried sample basis)
PI = % moisture regain of sample (dried sample basis);

then

$$PM = \frac{100(H - E)}{E} = \frac{100M}{D} \tag{6}$$

and

$$PI = \frac{100(G - F)}{F} = \frac{100I}{D} \tag{7}$$

LITERATURE CITED

1. American Society for Testing Materials, Designation C-177-45, "Standard Method of Test for Thermal Conductivity of Materials by Means of the Guarded Hot Plate."
2. National Bureau of Standards, Commercial Standard CS-42, "Structural Fiber Insulation Board;" and Commercial Standard CS-112-43, "Homogeneous Fiber Wallboard."
3. American Society for Testing Materials, Designation C-208-48, "Standard Specification for Structural Insulating Board Made from Vegetable Fibers."
4. Federal Standard Stock Catalogue, Section IV (Part 5), "Federal Specification for Fiber Board, Insulating. LLL-F-321b" (June 30, 1942, Amendment 1, Dec. 17, 1942).

Pentosans in Wood*

TAPPI Tentative Standard T 19 m-50

Pentosans are a part of the noncellulosic carbohydrates present in wood and consist chiefly of xylan and araban. The latter substances are converted to furfural with strong hydrochloric acid (HCl). In this method the furfural formed by the action of hot HCl on the pentosans in wood is distilled from the mixture in the manner prescribed by the A.O.A.C. (*1*). The distillates may be analyzed by either a gravimetric or a volumetric procedure. Pentosans in pulp (T 223 m) and paper (T 450 m) are determined by a volumetric method, but insufficient experimental data exists to ascertain the accuracy of the volumetric method for the determination of pentosans in all kinds of wood.

APPARATUS

1. A 300-ml. distilling flask, which in turn is connected to a long water-cooled condenser. A separatory or dropping funnel is mounted in the neck of the distilling flask. Rubber stoppers may be used to make the connections. It is preferable, however, to use a 300-ml. round-bottom flask with a No. 24/40 glass stopper, as shown in Fig. 1.
2. A 100-ml. graduated cylinder provided with a funnel for collecting the distillate.
3. Gooch crucibles.

REAGENTS

A. *Hydrochloric Acid*, 12% (sp. gr. 1.06). Dilute 307 ml. of C.P. conc. HCl (sp. gr. 1.18 to 1.19) to 1 liter with water.

B. *Phloroglucinol Solution.* Dissolve 11 grams of pure phloroglucinol in 300 ml. of warm 12% HCl and dilute to 1500 ml. with cold 12% HCl. If diresorcin is present, it will crystallize out after standing for a week. Filter immediately before using.

C. *Aniline Acetate Reagent.* Add acetic acid slowly to a mixture containing equal volumes of aniline and water until the liquid becomes clear after shaking.

TEST SPECIMEN

The test specimen shall consist of 1.5 grams of air-dry wood meal of known moisture content and so chosen as to represent the entire lot being tested (note 1). It shall be prepared for analysis in accordance with TAPPI Standard T 11 m. If extractive-free wood meal is used for analysis, it shall be prepared in accordance with T 12 m.

PROCEDURE

Place the accurately weighed sample in the 300-ml. distilling flask, together with a small piece of paraffin to eliminate foaming and a few glass beads or several pieces of recently ignited pumice stone to prevent bumping. Add 100 ml. of 12% HCl, place the flask on a wire gauze, connect with a condenser, heat gently at first with a Bunsen burner, and then regulate so as to distil over 30 ml. in each 10 minutes. More rapid distillation gives low results. Pass the distillate through a small filter paper before it enters the receiver, and keep the tip of the condenser as close as possible to the filter paper.

As soon as 30 ml. of distillate are collected, add another 30-ml. portion of 12% HCl from the separatory funnel in such a manner as to wash down particles adhering to the sides of the flask. Continue the distillation in this manner until 360 ml. of distillate are collected. To the total distillate add gradually with stirring 40 ml. of filtered phloroglucinol solution that has been prepared at least a week previously. The distillate will soon turn greenish black. Allow the solution to stand 16 hours. After this period the amorphous black precipitate of furfural phloroglucide will have settled to the bottom of the beaker. Test the clear supernatant liquid with aniline acetate paper. If a drop of the liquid gives a pink color with aniline acetate paper, the precipitation of the furfural is incomplete (note 2), in which case add a further amount of phloroglucinol solution and again allow the mixture to stand 16 hours.

Collect the furfural phloroglucide in a weighed Gooch crucible having a thick asbestos mat. Wash the precipitate with exactly 150 ml. of cold water in such a way that the water is not entirely removed from the crucible until the washing is completed. Dry the crucible for $2^1/_2$ hours in an air oven at 100 to 105°C. Cool in a stoppered weighing bottle and weigh. The increase in weight is considered to be furfural phloroglucide.

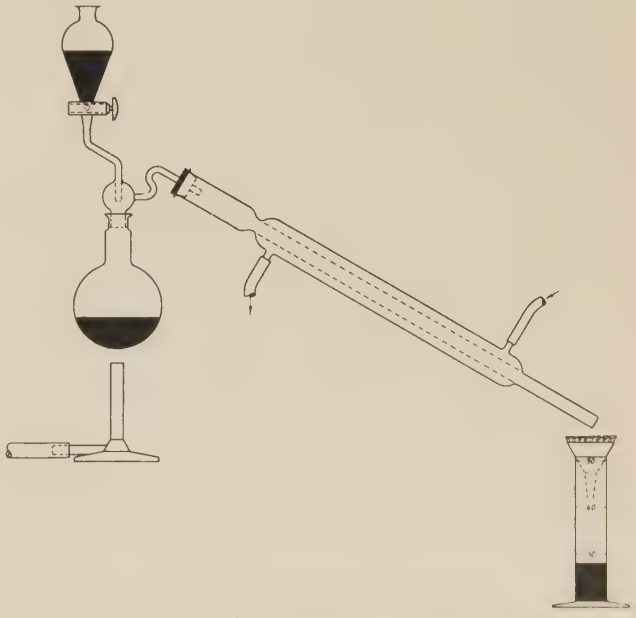

Fig. 1.—Apparatus for the determination of pentosans

* This method has been approved as a tentative standard by the Chemical Methods Committee. Criticisms are earnestly requested and should be sent to R. G. MacDonald, Secretary, Technical Association of the Pulp and Paper Industry, 122 E. 42nd St., New York 17, N. Y.

CALCULATION OF RESULTS

The weight of pentosans corresponding to the weight of furfural phloroglucide is found as follows:

$$\text{Pentosans} = (a + 0.0052) \times f$$

where

a = weight of furfural phloroglucide in grams

and

f = 0.895 if a is less than 0.03 gram
0.887 if a is between 0.03 and 0.3 gram
0.882 if a is more than 0.3 gram

Notes: (1) The weight of the sample should be such that the weight of phloroglucide obtained does not exceed 0.3 gram.
(2) The quantity of phloroglucinol used should be about double that needed for complete precipitation.
(3) The factor 0.0052 represents the weight of the phloroglucide that remains dissolved in the 400 ml. of acid solution.

REPORT

The pentosans, calculated from the furfural phloroglucide, shall be reported as a percentage of the weight of the moisture-free sample to one decimal place.

The results of duplicate determinations shall agree within 0.4.

LITERATURE CITED

1. Assoc. Off. Agr. Chemists, "Methods of Analysis," 6th ed. (1945), p. 412.

RECENT BOOKS

Ninth Graphic Arts Production Yearbook 1950. Colton Press, 468 Fourth Ave., New York. Fabricated leather $8^1/_2 \times 11$, 918 pages. $15.

Although several yearbooks and encyclopedias have been reviewed in this magazine this is the first time that a review of the Graphic Arts Production Yearbook has been reported. When *Tappi* was being launched in 1949 it became necessary for its staff to become acquainted with many complicated publication problems. Many questions were asked of the printing company production men. The latter were cooperative but undoubtedly felt that much time and effort could be saved if a reliable and ready source of information could be recommended. Their recommendation was a remarkable book issued each year by the Colton Press in New York, called the "Graphic Arts Production Yearbook."

Its eight major sections feature: Art and Photography; Photoengraving; Duplicate Plates and Ink; Paper; Printing Processes; Binding, Mailing and Finishing; Typography; and a Buyers Guide. Full page samples of 39 kinds of paper are included. The section on paper deals with such subjects as paper selection and sampling, printability, finishes, sizes and weights, estimating, etc. It is a valuable encyclopedia for all who need to know about printing and publishing. It is down to earth and abundantly illustrated with charts, tables, visualizers, drawings, and photographs.

Chemical Engineers Handbook. Edited by H. Perry, E. I. duPont de Nemours & Co., Inc. Revised 3rd Ed. New York. McGraw-Hill Book Co., 1950. Artificial leather $7^1/_4 \times 10$, 1942 pages. $15.

As will be noted by chemical engineers the Handbook has grown greatly in both number of pages and size of pages. Its 141 major contributors, each a specialist in his field has brought every important aspect of chemical engineering information up to date.

The subject headings, with a few additions are the same as those appearing in earlier editions. These cover physical and chemical data, and principles, heat transfer, flow of fluids, evaporation, gas absorption, humidification, drying, process control, materials of construction, fuels, materials handling, furnaces and kilns, power generation and transmission, plant location, electrical engineering, cost finding, etc.

The large influx of graduate chemical engineers into the industry during the past double decade attests to the recognition in the pulp and papermaking processes. The place of these men in the industry is well illustrated in the scope of the Handbook.

An Index of Nomograms. Compiled and edited by Douglas Payne Adams. Massachusetts Institute of Technology. 1950. John Wiley & Sons, Inc., New York. Cloth, $7^1/_2 \times 10$, 174 pages, $4.

This unusual index lists over 1700 published nomograms in well-known periodicals. The book is divided into two main parts; Index A—Key Words, and Index B—Master Index. By this means particular nomograms may be located by periodicals, date of issue, volume number, and page number. No nomograms are reproduced in this book. It is entirely an index to show quickly where needed nomograms may be found.

Physical Methods in Chemical Analysis, Vol. I. Edited by Walter G. Berl, Johns Hopkins University. 1950. Academic Press, Inc., New York. Cloth, 6×9, 664 pages. Illustrated $12.

As in the case of many books of this type there are a number of authors, including G. L. Clark, L. O. Brockway, R. A. Oetjen, J. H. Hibben, etc. Among the subjects covered are phenomena of x-rays, electron diffraction, spectro-photometry and colorimetry, Raman spectra, refractive index, and electron microscope.

A chapter by John A. Howsman of the American Viscose Corp., covers x-ray diffraction as applied to fibers and deals with the structure of fibers, the fiber diagram, orientation, and spiral fiber characteristics.

Colloid Science II. Edited by H. R. Knott, President, Central National Council for Applied Scientific Research in the Netherlands. Elsevier Publishing Co., New York. 1949. Cloth $6^1/_2 \times 10$, 753 pages. $11.50.

Volume 1 of this series dealt with irreversible systems. The present volumes cover reversible systems, macramolecular and association colloids. These terms are used as an improvement, based on the thermodynamic justification, of the older terms lyophobic and lyophilic systems. It is the intent of this work to be a guide to the domain of colloid science and a stimulus for research in this field. The principal subjects covered in Vol. II are formation and structure of macramolecules, thermodynamics of long-chain molecules, the physical properties of randomly kinked long-chain molecules, molecular weight, sols without electrylyto character, flocculation, complex colloid systems, gels, association colloids, and morphology of coacervates. As in other works of a similar nature each subject is handled by an authority in that field.

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Calendar of TAPPI Meetings

NATIONAL

Second Testing Division Conference. Eastman Kodak Co. plant, Rochester, N. Y., September 13–15, 1950.

- General Chairman—James d'A. Clark, Longview, Wash.
- Fibrous Materials Testing Committee—E. R. Schafer, Forest Products Laboratory, Madison, Wis., Chairman.
- Nonfibrous Materials Testing Committee—J. P. Casey, New York State College of Forestry, Syracuse, N. Y., Chairman.
- Wax Testing Committee—A. M. Heald, Marathon Corp., Menasha, Wis., Chairman.
- Pulp Testing Committee—L. V. Forman, Institute of Paper Chemistry, Appleton, Wis., Chairman.
- Optical Properties Committee—A. H. Croup, Hammermill Paper Co., Erie, Pa., Chairman.
- Microscopy Committee—C. E. Brandon, Aetna Paper Co., Dayton, Ohio, Chairman.
- Paper Testing Committee—A. K. Warner, Skinner & Sherman, Inc., 246 Stuart St., Boston, Mass., Chairman.
- Container Testing Committee—W. J. Balster, Don L. Quinn Laboratories, Chicago, Ill., Chairman.
- Chemical Methods Committee—Milton Fillius, Eastman Kodak Co., Chairman.
- Packaging Materials Testing Committee—L. E. Simerl, Marathon Corp., Menasha, Wis., Chairman.

Fifth Engineering Conference of the Pulp and Paper Industry. Netherland Plaza Hotel, Cincinnati, Ohio, October 2–5, 1950.

- General Chairman—G. H. Pringle, Mead Corp., Chillicothe, Ohio.
- Steam and Power Committee—C. J. Sibler, West Virginia Pulp and Paper Co., New York, N. Y., Chairman.
- Materials Handling Committee—A. P. Schnyder, Ebasco Services, Inc., New York, N. Y., Chairman.
- Drying and Ventilating Committee—M. L. Barker, Beckett Paper Co., Hamilton, Ohio, Chairman.
- Engineering Research and Machine Design Committee—P. H. Goldsmith, Pusey & Jones Corp., Wilmington, Del., Chairman.
- Mill Design and Engineering Aspects Committee—Alvin H. Johnson, New York, N. Y., Chairman.
- Hydraulics Committee—K. J. Mackenzie, Eastman Kodak Co., Rochester, N. Y., Chairman.
- Mill Maintenance and Materials Committee—H. F. Parker, New York and Pennsylvania Co., Lock Haven, Pa., Chairman.
- Electrical Engineering Committee—A. S. Goodrich, Hammermill Paper Co., Erie, Pa., Chairman.
- Chemical Engineering Committee—J. R. Lientz, Union Bag & Paper Corp., Savannah, Ga., Chairman.
- Data Sheets Committee—H. J. Perry, Lockwood Trade Journal Co., New York, N. Y., Chairman.

Plastics Conference. New York State College of Forestry, Syracuse, N. Y. October 19–20, 1950.

Alkaline Pulping Conference. George Washington Hotel, Jacksonville, Fla., October 25–27, 1950.

Fibrous Agricultural Residues Conference. Northern Regional Research Laboratory, Peoria, Ill., November 12–13, 1950.

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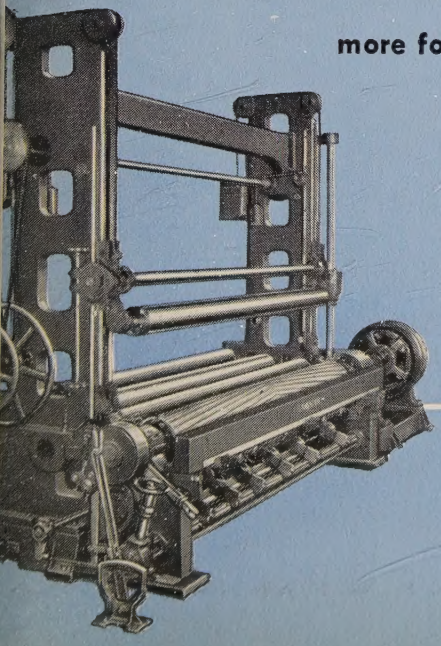

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